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PRECISION EXTRUSION OF HOLLOW TURBINE VANES OF OXIDE DISPERSION--ETC(U)

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PRECISION EXTRUSION OF HOLLOW TURBINE VANES OF OXIDE DISPERSION STRENGTHENED NICKEL CHROMIUM ALLOY BY THE FILLED BILLET METHOD

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CINCINNATI, OHIO 45215

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AIR FORCE MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

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This technical report has been reviewed and is approved for publication.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Precision extrusion of hollow vanes of oxide dispersion strengthened nickel chromium alloys was demonstrated on subscale specimens using a modification of the filled billet method. The desired <100> microstructure was attained after heat treatment of extruded vanes. Stress rupture life at 2000°F at 11 ksi exceeded 100 hours. A full scale processing sequence based on subscale results is proposed.

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FOREWORD

This Final Technical Report covers all work performed under Contract F33615-74-C-5150 by the Polymet Corporation, Cincinnati, Ohio, from 13 March 1974 to 15 November 1975. The report was released by the author on 15 March 1976.

This contract was initiated under Project 7351, "Metallic Materials", Task 735101, "High Temperature Metals", Work Unit 73510172. The work was performed under the technical direction of Mr. William T. O'Hara of the Metals and Ceramics Division of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

Mr. James G. Hunt and Mr. George J. Wile of Polymet Corporation were responsible for the management and execution of the program. Mr. Hunt served as the principal investigator.

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SUMMARY

The precision extrusion of hollow vanes of dispersion strengthened nickel chromium alloy was investigated in a three-phase program, which successfully applied a modification of the filled billet extrusion method.

In the first phase the general relationship between thermomechanical processing conditions and recrystallization behavior were determined by conventional filled billet extrusion. Extrusion parameters included primarily preheat temperature, reduction ratio and die entry angle. In this portion of the work, a microstructure was obtained which was only partly recrystallized.

In the second phase, extrusion conditions which caused recrystallization entirely to the desired <100> microstructure were determined and a necessary modification of the filled billet method incorporating these conditions was verified. The revised method produced vane material with acceptable 2000°F stress rupture life.

In the third phase, methods of producing preforms were evaluated and then double hollow turbine vanes with prescribed properties were extruded by the revised filled billet method. The extrusions were evaluated and found to have recrystallized properties equal or superior to recrystallized sheet of the same alloy. A production process, based on the subscale work, was proposed for full size hollow turbine vanes.

I. INTRODUCTION

1. Program Background

Increasing thrust and efficiency of advanced turbine engines generally demand increasing temperatures in the turbine section. Higher vane temperatures impose the need for higher performance materials.

Oxide dispersion strengthened nickel and cobalt alloys have demonstrated satisfactory performance in advanced test engines. Because of its advanced development, the alloy Ni-20Cr-2ThO_2 has been initially chosen for the F101 engines for the B1 bomber, and is therefore the alloy selected for experimentation in this program.

The design of the high pressure nozzle vanes for this engine involves two hollows, parallel with the leading and trailing edge of the vane. At this time the vanes are produced by machining them from a rectangular extrusion made by an exacting thermomechanical processing sequence, followed by directional recrystallization. In the manufacture of vanes, the weight of scrap is now almost ten times the weight of the finished vanes. The cost of the vanes is therefore quite high. Extensive use of Ni-20Cr-2ThO_2 alloy, and similar materials, could be increased by significant increases in materials yield.

The method of conventional shaped extrusion could produce Ni-20Cr-2ThO_2 alloy bars with less envelope for machining. However, it cannot produce the double hollow in the vane, nor can it produce the thin trailing edge, and, at the same time, maintain the necessarily uniform temperature profile across the extruding section during deformation. This uniformity is necessary since it induces the desired recrystallized microstructure and consequent mechanical properties. Conventional extrusion, therefore, can only provide marginal cost reduction.

A special extrusion technique, called the filled-billet method, has been successfully used to produce precise, shaped, hollow sections of various high temperature alloys.

It incorporates the following four basic steps which are sketched in Figure 1:

- producing a shaped hollow preform of an alloy,
- canning the preform in a filler material to comprise a billet,
- coextruding the preform and filler material, and then
- removing the filler material.

In the canning process the shaped preform is inserted into the filler having a shaped inside to fit the preform and a cylindrical outside to fit extrusion tools. The material comprising the filler is chosen to match as closely as possible the extrusion characteristics of the preform, and, at the same time, be reasonable in cost, easy to shape, and readily available.

The preform shape is related mathematically to the extruded shape. If the extrusion reduction is R, then the finished length is theoretically R times the preform length, and any given dimension on the preform's transverse cross section is $\sqrt{R}$ times the corresponding dimension of the finished section. When the filler material has about the same "relative stiffness" as the preform, and the billet components are not porous, so that significant dimensional changes in the billet do not occur, the actual dimensions of the extruded section can often be obtained within about one percent of the theoretical dimensions.

The extrusion reduction is the ratio of the transverse cross sectional areas of the billet and the extruded rod. Relative stiffness is a measure of the deformation resistance of a specific material at a given temperature. When two materials are coextruded, as in the filled billet method, the stiffness of the constituents of the coextrusion should be similar. The relative stiffness, k, of a material at a given temperature is given by the relation $k = F/A \ln R$, where F is the extrusion force, A is the transverse cross section of the billet, and $\ln R$ is the natural logarithm of the reduction ratio.

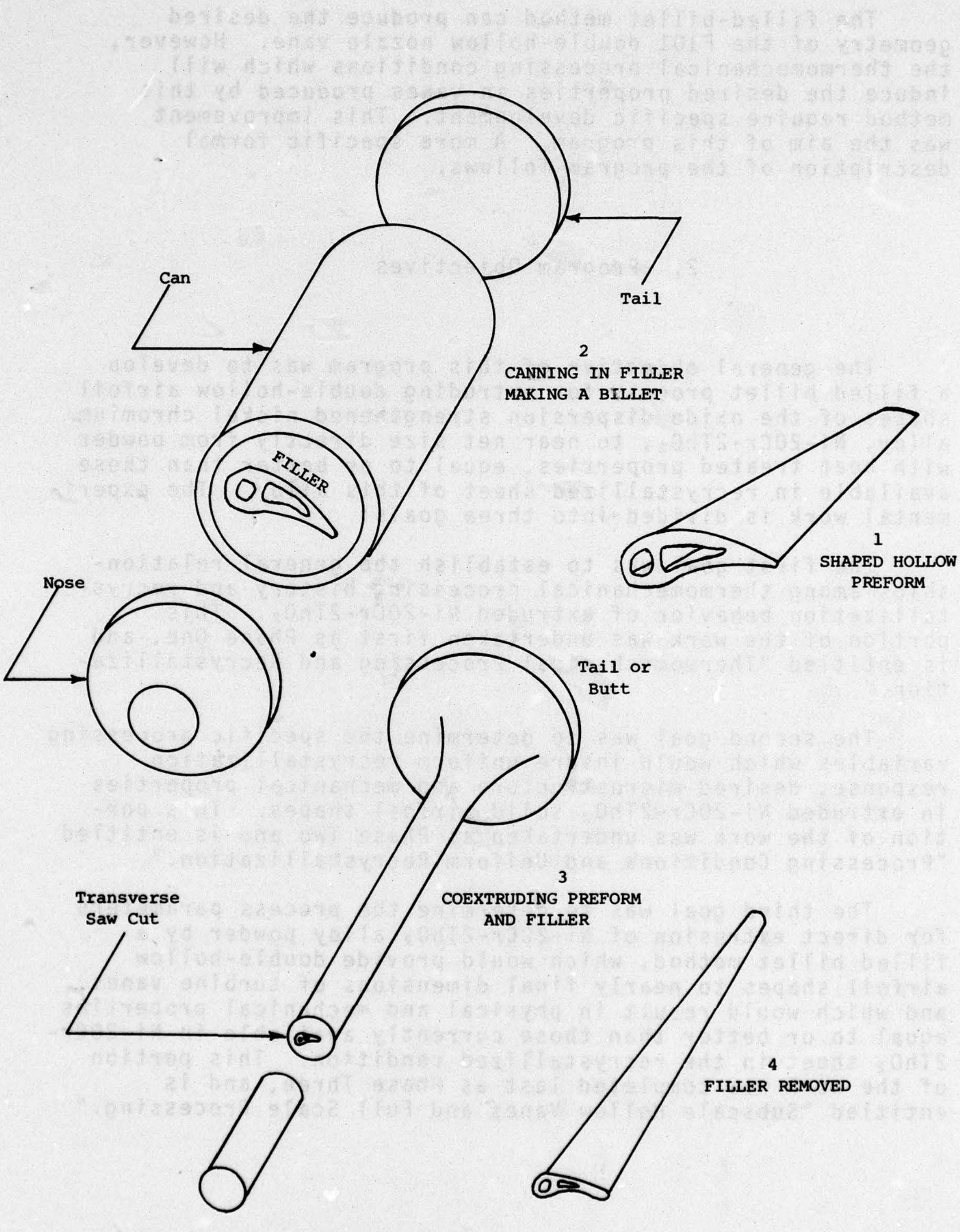


FIGURE 1. SKETCH OF CONCEPT OF FILLED BILLET METHOD

The filled-billet method can produce the desired geometry of the F101 double-hollow nozzle vane. However, the thermomechanical processing conditions which will induce the desired properties in vanes produced by this method require specific development. This improvement was the aim of this program. A more specific formal description of the program follows.

2. Program Objectives

The general objective of this program was to develop a filled billet process for extruding double-hollow airfoil shapes of the oxide dispersion strengthened nickel chromium alloy, Ni-20Cr-2ThO_2, to near net size directly from powder with heat treated properties, equal to or better than those available in recrystallized sheet of this alloy. The experimental work is divided into three goals:

The first goal was to establish the general relationships among thermomechanical processing history and recrystallization behavior of extruded Ni-20Cr-2ThO_2. This portion of the work was undertaken first as Phase One, and is entitled "Thermomechanical Processing and Recrystallization."

The second goal was to determine the specific processing variables which would insure uniform recrystallization response, desired microstructure and mechanical properties in extruded Ni-20Cr-2ThO_2 solid airfoil shapes. This portion of the work was undertaken as Phase Two and is entitled "Processing Conditions and Uniform Recrystallization."

The third goal was to determine the process parameters for direct extrusion of Ni-20Cr-2ThO_2 alloy powder by a filled billet method, which would provide double-hollow airfoil shapes to nearly final dimensions of turbine vanes, and which would result in physical and mechanical properties equal to or better than those currently available in Ni-20Cr-2ThO_2 sheet in the recrystallized condition. This portion of the work was completed last as Phase Three, and is entitled "Subscale Hollow Vanes and Full Scale Processing."

II. PHASE ONE:

THERMOMECHANICAL PROCESSING AND RECRYSTALLIZATION

1. Scope

The general relationships between various extrusion conditions and recrystallized microstructure were investigated by preparing and extruding twenty billets and then metallographically examining selected recrystallized extruded sections. The thermomechanical processing variables investigated were temperature, reduction ratio and degree of shearing, caused by changing die entry angle.

2. Procedure

The following steps were involved:

Isostatic Pressing: Fifty pounds of Ni-20Cr-$2ThO_2$ alloy powder were procured from Sherritt Gordon Mines Ltd. About 20 pounds of the powder were isostatically pressed at 33 ksi at Sylvania in Towanda, into three cylindrical bars about 1.2 inches in diameter and 20 inches long. The relative density was calculated to be about 65 percent.

Canning: The pressed cylinders above were machined into filled-billet cores approximately five inches long and in most cases one inch in diameter. A .050 inch groove was machined around the core at one inch intervals along its length. The cores were concentricly canned in low carbon steel billets by TIG welding. A sketch of the billet design is shown in Figure 2. This design was intended to produce an extruded specimen with a taper, formed by varying the reduction in area. Following evacuation to about five microns, the billets were sealed at one end of the billet, either by electron beam welding a small hole, or peening shut an evacuation tube.

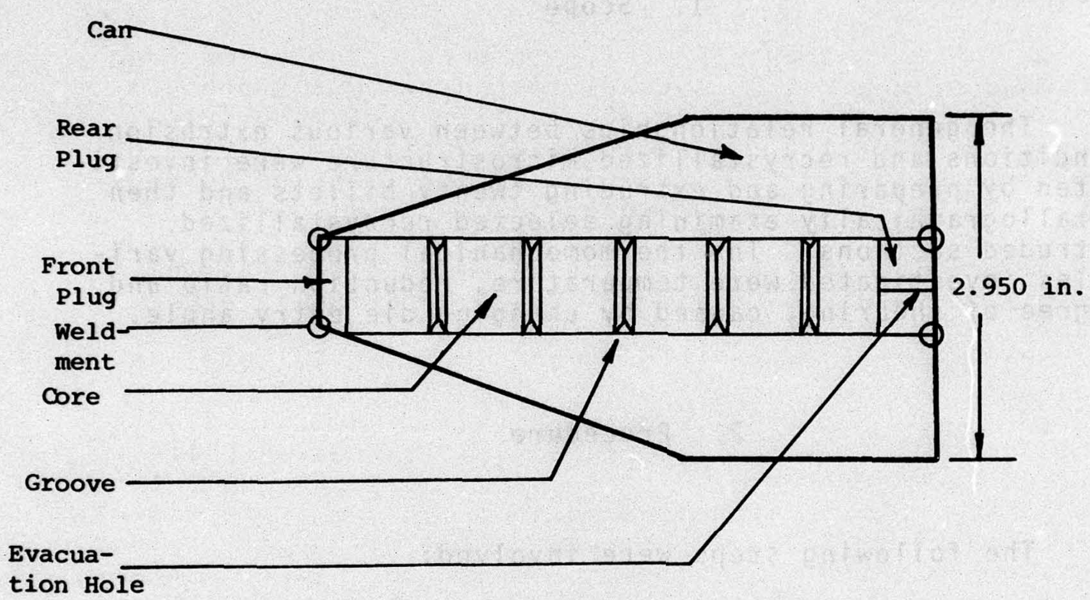


FIGURE 2. SKETCH OF BILLET DESIGN IN PHASE ONE

Alloy cores were canned in steel billets with this design to produce rods which had reductions in area between about five and 25 times.

Preheating: After precoating the billets with Polygraph, a commercial oxidation retardant and lubricant, the billets were heated and soaked at temperature, between 1650° and 2250°F, and between 1.5 and 2.5 hours in a Globar furnace in oxidation resistant metal retorts in an air atmosphere.

Extruding: The billets were transferred from the furnace in about 15 seconds to the extrusion press, the 700 ton Lombard machine at the Air Force Materials Laboratory, operated by Westinghouse employees. The tooling used is illustrated in Figure 3. Zirconia coated H-13 dies and graphite follow blocks were used.

The variables investigated were preheat temperature (between 1650° and 2250°F), reduction in area (between 5 and 25 times), die entry angle (30° and 90°), and core relative density (65 % and 100 %). Fully dense cores were made by compacting some of the hydrostatically pressed cores, referred to previously, at 100 tsi, 2100°F for 10 seconds in billets designed like extrusion billets, and then decanned by machining. Ram speed, can wall thickness, transfer time, lubrication, tooling temperature were held constant in these experiments. All of the extrusions were air cooled. The conditions for various experimental billets are shown in Table 1.

Before samples of the extrusions were taken for metallographic examination, the excess steel from the front and rear of the rod was cropped off by sawing. The steel was removed from the rest of the rod by pickling in dilute nitric acid solution. The appearance of the extrusions is sketched in Figure 4. The dimensions of the extrusions were measured and recorded. The reduction in area for a given position on the tapered extruded core was then calculated by determining the square of the diameter ratio between the upset (or consolidated diameter) and the extruded diameter. The upset diameter was found by measuring the decanned core of several typical upset billets (Numbers 16-20 in Table 1) used to produce dense cores referred to above. The surface quality of the extrusions also was recorded.

Recrystallizing: All of the specimens selected for metallographic examination in Phase One were recrystallized in air in a Globar furnace at 2450°F for 2 hours.

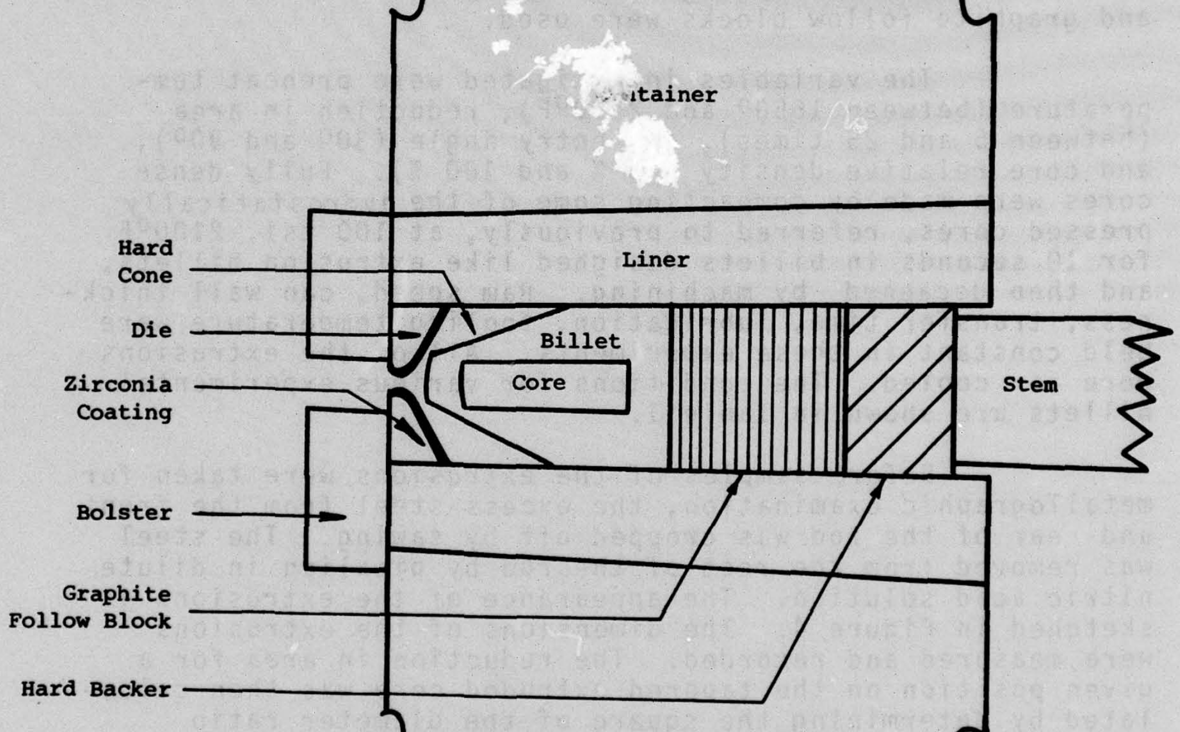


FIGURE 3. SKETCH OF TOOLING DESIGN IN PHASE ONE.

Most of the extrusions for Phase One were made in this tooling which produced cores with varying reductions in area.

TABLE 1

EXTRUSION CONDITIONS AND RESULTS IN PHASE ONE[1]

Conditions				Results					
				Texture at 5X			Texture at 25X		
Billet or Extrusion No.	Core Density (%)	Die Angle (°)	Preheat Temp (°F)	<100> Outer Rim	<110> Inner Ring	un Rx Central Core	<100> Outer Rim	<110> Inner Ring	un Rx Central Core
1	100	90	1650	none	none	very large	thin	none	large
2	100	90	1700	--	--	very large	thin	none	large
3	100	90	1800	--	--	very large	thin	none	small
4	100	90	1850	thin	none	very large	thick	none	small
5	65	90	1850	--	--	--	thick	none	small
6	65	30	1900	none	none	very large	thick	none	small
7	65	30	1950	none	none	very large	thick	none	small
8	100	90	2000	none	none	very large	thick	none	very small
9	65	90	2000	--	--	--	thick	none	very small
10	65	30	2000	--	--	--	thick	none	small
11	65	30	2050	none	thin	large	thick	thin	small
12	65	30	2100	--	--	--	thick	thin	small
13	65	30	2150	--	--	--	thick	thin	small
14	65	30	2200	--	--	--	thin	thick	large
15	65	30	2250	--	--	--	thin	thick	large

Notes: <110> = presumed for non <100>   un Rx = not recrystallized

Other Conditions:

Reductions	5 to 25X	Preheat time	1.5 - 2.0 hours
Ram Speed	130 $\pm$ 15 inches/minute	Lubrication	Fisk 604D
Can Wall	1.0 inch	Tool Temperature	500°F
Transfer Time	15 $\pm$ 5 seconds	Cooling Method	Air

[1]Billets 16-20 were designed like others but were only upset at 2100°F and 100 tsi, and used for the dense cores in Billets 1, 2, 3, 4, and 8.

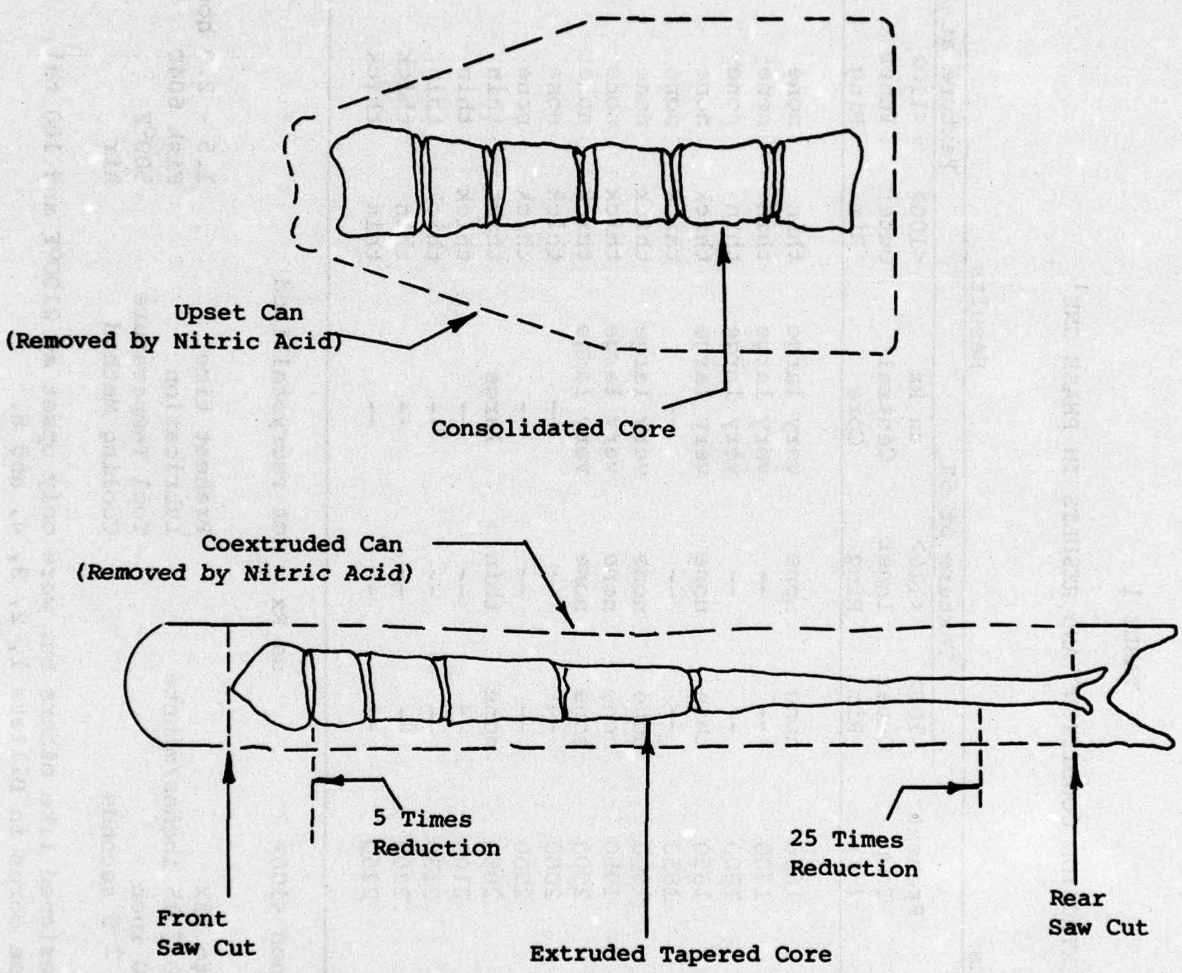


FIGURE 4. SKETCH OF CORES AND RODS OF PHASE ONE.

The billet design shown in Figure 2 produced rods with tapered cores with reductions in area between about five and 25 times reduction, as sketched in the lower view. The upper view shows the presumed condition of the core at upset before extrusion. In billets 16 through 20, the cores were only compacted to this condition, followed by can removal.

The specimens were loaded at 2200°F on an alumina refractory, and heated to 2450°F in 30 minutes. After two hours, the specimens were removed from the furnace and air cooled.

Metallographic Examination: The specimens were cast in epoxy resin with alumina filler for maximum edge retention. After polishing with successively finer silicon carbide papers, the finest being 600 grit, the specimens were polished with 15 and then 6 micron diamond polishing compound. Final polishing was completed with Linde B submicron alumina. Following etching in dilute Kalling's reagent, the samples were viewed on a Bausch and Lomb Palchot metallograph at Metcut Research Associates. The specimens were examined for the extent and type of grain structure.

3. Results

The dimensions of the extruded samples made from dense cores indicated that relatively uniform streamlined flow had taken place during extrusion, being within five percent of that anticipated from the reduction ratio calculated from tooling sizes. (The reduction ratio for a solid, uncanned billet is equal to the square of the ratio of the liner diameter to the die diameter.) Dimensions of extrusions made from undense cores were only meaningful in that they provided a basis for establishing the reduction that had produced the tapered extruded samples.

The surface quality of all extruded sections resembled a fine ground surface. Increasing reductions were found to lead to increasing smoothness.

Since the microstructure of unheat-treated extrusions appeared to be very similar for samples made by different processing conditions, metallographic examination of this type of specimen was carried out on a few samples and then discontinued. However, the microstructure of heat-treated extrusions showed a close correlation with the thermo-mechanical processing conditions of extrusion temperature and reduction. The results (see Table 1 and Figures 5 and 6) can be summarized as follows:

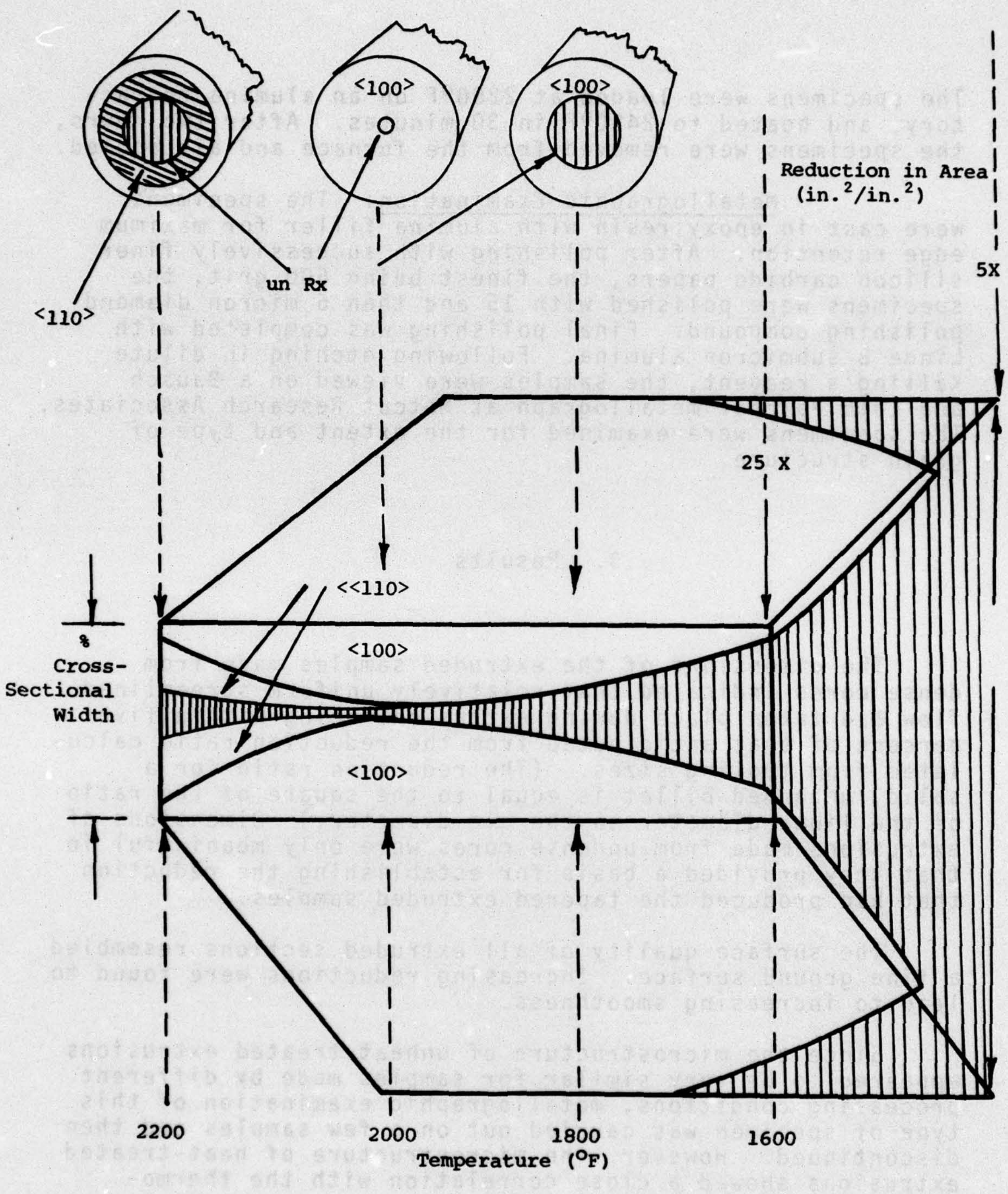
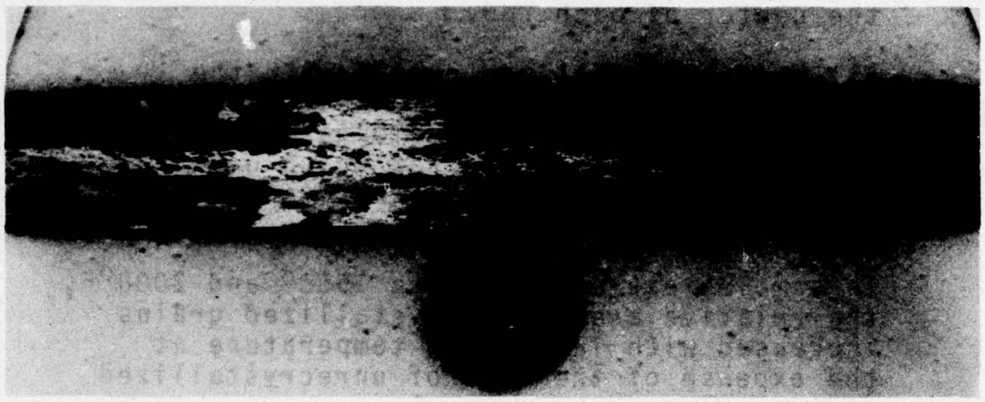


FIGURE 5. PLOT OF MICROSTRUCTURES OBTAINED IN PHASE ONE

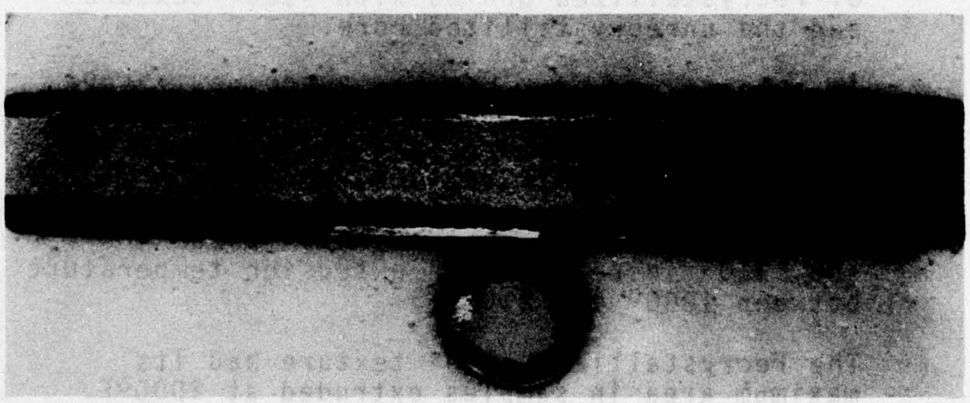
A representation of the recrystallized microstructures is schematically plotted for various extrusion conditions.



Longitudinal

Transverse

Extruded 1650° - 2000°F



Longitudinal

Transverse

Extruded 2050° - 2250°F

FIGURE 6. PHOTOMICROGRAPHS OF REPRESENTATIVE RODS IN PHASE ONE

Cores extruded at 25 times reduction always had nonrecrystallized material, an outer rim of <100> texture, and, when extruded above 2050°F, an inner rim of non <100> texture.

5X Magnification

In the samples representing extrusion at 25 times reduction and preheat temperatures between 1650° and 2250°F:

- All had some unrecrystallized material at the center of each rod.
- All had recrystallized grains with the desired <100> crystallographic texture that were very large and elongated in the extrusion direction in area which began at the surface and extended inward toward the center of each rod.
- In samples extruded between 1650° and 2000°F, the relative area of recrystallized grains increased with increasing temperature at the expense of the area of unrecrystallized grains.
- All samples extruded between 2050° and 2250°F had recrystallized grains with <110> texture forming an inner ring between the outer rim of recrystallized grains with <100> texture and the unrecrystallized core.
- In samples extruded between 2050° and 2250°F, the unrecrystallized core grew in size with increasing temperature at the expense of the area of recrystallized <100> texture.
- The recrystallized grain size of both <100> and <110> increased with extrusion temperature between 1800° and 2100°F.
- The recrystallized <100> texture had its maximum area in samples extruded at 2000°F.
- The unrecrystallized region had its minimum area in samples extruded at 2000°F.
- Recrystallized grains with <110> orientation were associated with extrusions carried out at 2050°F and above.

In the samples extruded at five times reduction, the recrystallized microstructures (compared to their higher reduction counterparts, carried out at the same temperature) indicated consistent trends:

- The areas of unrecrystallized material, which generally increased with increasing temperature, were larger at all temperatures.
- Above 2050^{o}F, and with increasing temperature, the areas of recrystallized grains with <110> texture became larger.

Initial core density had apparently undetected effect on recrystallized microstructure. This conclusion is based on the observation that the dense core of Billet 8, and 65 % dense powder core of Billet 9, both extruded at 2000^{o}F and 25 times reduction, produced the same microstructural pattern and attendant areas, a rim of recrystallized <100> grains around a very small unrecrystallized core. However, die entry angle had a small but perhaps important effect when the core was extruded at 25 times and 2000^{o}F. The unrecrystallized core of Billet 9, extruded through a die with 90^{o} included angle, was significantly smaller than that of Billet 10 extruded through a die with 30^{o} included angle.

4. Discussion

Among the various extrusion conditions tried, the ones used to process Billets 8 and 9 gave the best results because the extrusions made from these billets contained the minimum amount of unrecrystallized grains and, at the same time, no detectable amount of <110> recrystallized grains.

As previously noted herein, extrusion 10 was made like 8 and 9 except with decreased die angle, and had, after recrystallization, a larger amount of unrecrystallized material. This result suggests that the amount of unrecrystallized grains present in extrusions 8 and 9 could be decreased by increasing the die entry angle beyond 90^{o}. This change would increase the amount of shearing and thereby possibly impart greater energy into the material for recrystallization. But, this increased energy for complete recrystallization to the desired <100> microstructure might also be induced individually or in combination by higher extrusion reductions, faster extrusion speeds, each and all carried out with minimum increase in temperature.

The importance of shear in developing the desired <100> microstructure might be indicated by the observation that the recrystallized 100 grains in the extrusions, produced in this portion of the work, always made their appearance on the outside rim of the sample. This is the region of maximum shear during extrusion. Unrecrystallized material appeared at the center of the sample, the region of minimum shear.

Apparently, recrystallized <100> grains could be induced even at the center of the sample if the amount of shear could be increased in that region.

This might be accomplished in two ways. One is to increase the amount and, possibly, the depth of shear in the extrusion by increasing the die entry angle, as previously noted herein. The other is to increase the depth of shear, by changing the configuration of the die from a circle to an ellipse or near rectangle. Then, if the depth of sufficient shear to induce the desired <100> microstructure is almost deep enough in round extrusion, it may extend to the center of the bar in elliptical or rectangular extrusion.

Experiments designed to investigate these and other relationships are described in Phase Two.

III. PHASE TWO:

PROCESSING CONDITIONS AND UNIFORM MICROSTRUCTURE

1. Scope

First, work in Phase Two involved the determination of processing conditions which would lead to uniform <100> recrystallization. Second, those extrusions that were successfully recrystallized to this microstructure were evaluated by tensile and stress rupture tests.

Two groups of extrusions were performed. In the first group, we sought extrusion conditions that would improve the best result in Phase One and eliminate the small amount of unrecrystallized material present among the <100> grains. In the second group, we sought extrusion conditions that would modify the typical duplex microstructure and obtain greater understanding of the relation between processing conditions and recrystallization.

2. Procedure

The processing conditions for the first groups of extrusions, Numbers 21 through 24, had numerous modifications over those for extrusion 9, the best result in Phase One. These are tabulated in Table 2 and described below. The billet design is sketched in Figure 7.

An elliptical die with a major to minor diameter ratio of 2.5 to 1.0 and a die entry angle of 90^{o} was used for Extrusion 21. This ratio is referred to as width/thickness ratio or W/T.

A round die with the entry angle increased from 90^{o} to 120^{o} was used for extrusion 22.

A round die with a higher reduction ratio of 36 times was used for extrusion 23, and, finally,

TABLE 2[1]

EXTRUSION CONDITIONS AND RESULTS IN PHASE TWO

		Conditions									Results		
		Preform				Die					<100>	<110>	un Rx
Billet or Extrusion No.	Variable Changed	Shape	Position	Pre-Extrusion	Red X	W/T x/1	L (°)	Speed (in./min.)	Temp (°F)		Outer Ring	Inner Ring	Central Core
				FIRST GROUP									
9	Control	Round	Central	None	25	1.0	90	120	2000		Thick	None	Very small
21	W/T Ratio	Round	Central	None	25	2.5	90	120	2000		Entire	None	None
22	Die Angle	Round	Central	None	25	1.0	120	120	2000		Thick	None	Very small
23	Reduction	Round	Central	None	36	1.0	90	120	2000		Thick	None	Very small
24	Core Shape	Vane	Central	None	10	2.5	90	120	2000		Entire	None	None
				SECOND GROUP									
13	Control	Round	Central	None	25	1.0		120	2150		Thick	Thin	Small
25	W/T Ratio	Round	Central	None	25	3.0	30	120	2150		Entire	None	None
26	Die Angle	Round	Central	None	25	1.0	120	120	2150		None	Thin	Large
27	Reduction	Round	Central	None	36	1.0	30	120	2150		Thick	Thin	Small
28	Reextrusion	Round	Central	8X	3	1.0	30	120	2150		Thick	Thin	Small
29	Reextrusion	Round	Central	25X	3	1.0	30	120	2150		Thick	Thin	Small
30	Speed	Round	Central	None	3	1.0	30	40	2150		None	None	Entire
31	Eccentricity	Round	Side	None	3	1.0	30	120	2150		Thick	Thin	Eccentricity
32	Eccentricity	Round	Edge	None	25	1.0	30	120	2150		Thick	Thin	Eccentricity

Notes: W/T Ratio - Width Thickness Ratio   un Rx - not recrystallized

Other Conditions:

Can Wall	1.0 inch except Billet 28	Tool Temperature	500°F
Transfer Time	15 ± 5 seconds	Cooling Method	Air
Lubrication	Fisk 604D	Preheat Time	1.5 - 2.0 hours
Preheat Temperature	2150°F except Billet 28 which had 2000°F preheat		

[1]Billet 9 and 13 from Phase One included for comparison.

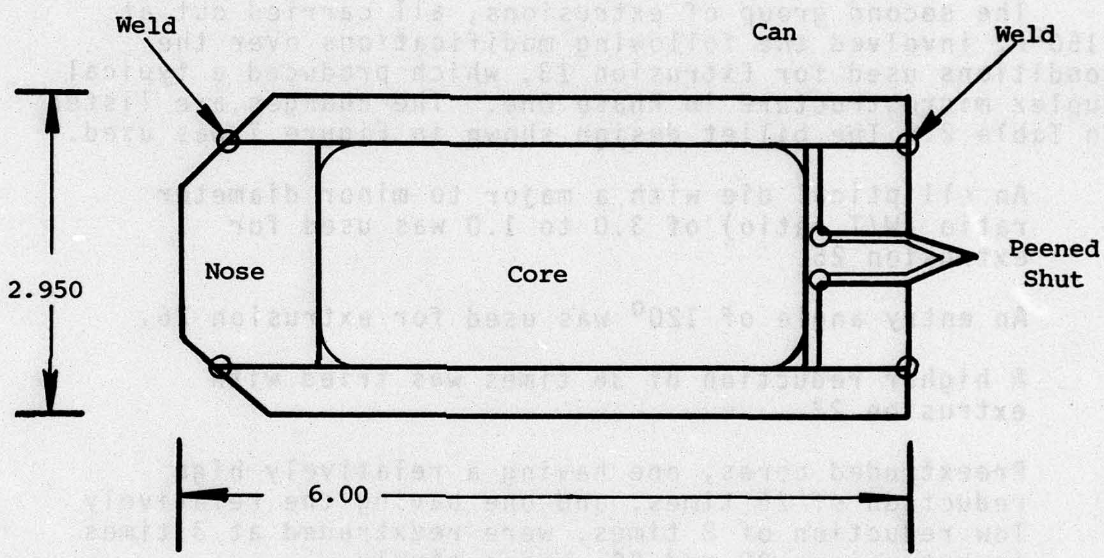


FIGURE 7. SKETCH OF BILLET DESIGN IN PHASE TWO

The elliptical die, used for Extrusion 21 (above), and a vane shaped core was tried in extrusion 24. The core was made by band sawing a vane shape from a solid isostatically pressed blade. The inside surface of the can also was shaped by band sawing. A photograph of the core is seen in Figure 8. The preheated billet, containing this core, was placed in the extrusion liner so that an imaginary plane passing through the leading and trailing edge was parallel with the major axis of the die opening.

The second group of extrusions, all carried out at 2150°F, involved the following modifications over the conditions used for Extrusion 13, which produced a typical duplex microstructure in Phase One. The changes are listed in Table 2. The billet design shown in Figure 7 was used.

An elliptical die with a major to minor diameter ratio (W/T ratio) of 3.0 to 1.0 was used for extrusion 25.

An entry angle of 120° was used for extrusion 26.

A higher reduction of 36 times was tried with extrusion 27.

Preextruded cores, one having a relatively high reduction of 25 times, and one having the relatively low reduction of 8 times, were reextruded at 3 times in extrusions 28 and 29, respectively.

The ram speed was decreased from 120 to 40 inches/minute for extrusion 30.

Cores were extruded with increasing degrees of eccentricity in extrusions 31 and 32.

3. Results

Microstructural

After coextruded steel was removed from the extrusions, samples about one-inch long were cut from the center

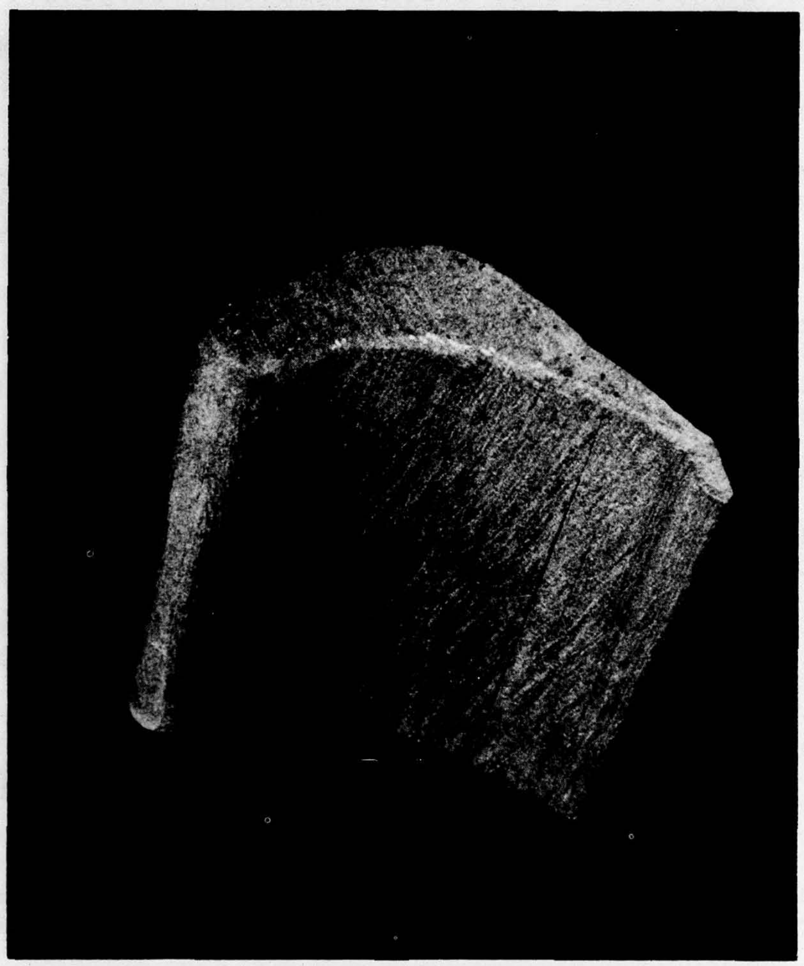


FIGURE 8. PHOTOGRAPH OF SOLID VANE PREFORM IN PHASE TWO

This preform was sawed from a compact produced by isostatic pressing at 33 ksi (Scale about 1.5X).

of each extrusion. The samples were furnace recrystallized at 2450°F for two hours in the same manner as that used in Phase One. Directional recrystallization also was performed on samples from extrusions 21, 24 and 25. This technique and results are reported in Phase Three.

In the first group of extrusions, done at 2000°F, the change to an elliptical die, caused complete recrystallization of the core to pure <100> in both the round core extrusion 21 and vane shaped core in extrusion 24. A photomicrograph of a recrystallized sample taken from extrusion 24 is shown in Figure 9 (bottom).

However, both increased die entry angle in extrusion 22, and increased reduction in extrusion 23, produced no detectable change in microstructure compared to extrusion 9, the control.

In the second group of extrusions, done at 2150°F, the elliptical die produced a pure <100> recrystallized microstructure in extrusion 25 as shown in Figure 9 (top). The increased die entry angle for extrusion 26 enlarged the area of unrecrystallized material compared to extrusion 13, the control.

Increased reduction of 36 times obtained in one extrusion in extrusion 27, or in two extrusions in extrusions 28 and 29, caused only a small decrease in unrecrystallized material, compared to extrusion 13. Decrease in ram speed to 40 inches/minute caused extrusion 30 to have an entirely unrecrystallized microstructure.

Increasing billet core eccentricity caused an opposite and proportional movement in the location of the unrecrystallized region in the samples from extrusions 31 and 32. As the core was moved toward the side of the billet, the unrecrystallized region tended to move toward the opposite side of the sample. Apparently, the unrecrystallized core tended to remain concentric with the central axis of the extrusion.

Mechanical

The extrusions which on heat treatment at 2450°F for two hours produced a fully recrystallized <100> microstructure were mechanically tested at elevated temperature.

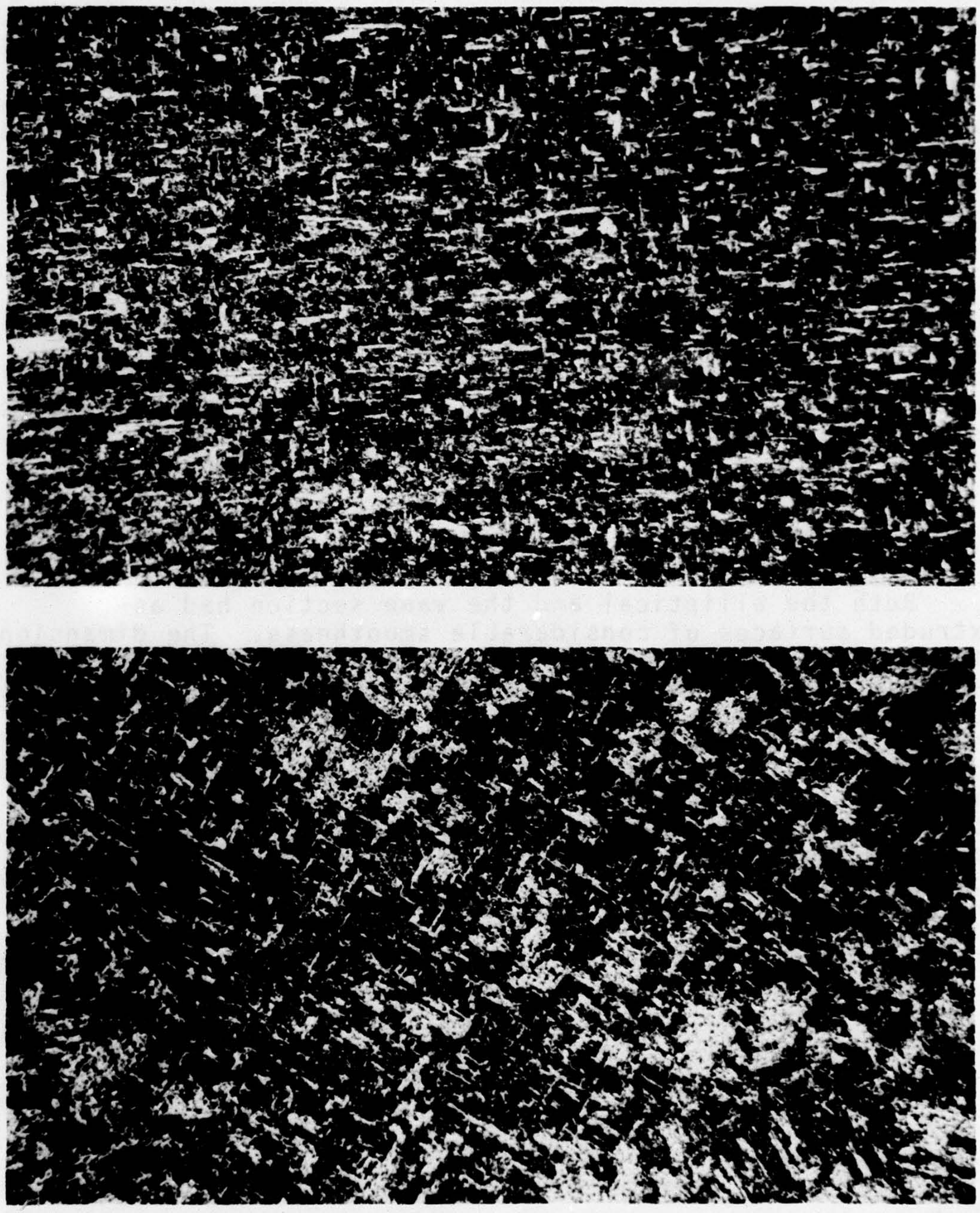


FIGURE 9. PHOTOMICROGRAPHS OF SELECTED SECTIONS IN PHASE TWO

Top: 2150°F, 25X, 3.0 to 1 W/T Ratio (Extrusion 25)

Bottom: 2000°F, 10X, 2.5 to 1 W/T Ratio (Extrusion 24)

Magnification 300X. Transverse section.

A heat treated sample from the elliptical extrusion carried out at 2000°F (Billet 21) was tested for stress rupture life at 2000°F and 10 ksi. After 125 hours, a portion of the grips failed and the test was discontinued. A 2000°F tensile test showed a UTS of 12.7 ksi, a 0.2 % YS of 8.9 ksi, and an elongation of 29 %.

The solid vane extrusion from Billet 24 also was tested for 2000°F stress rupture life. After 240 hours at 10 ksi, the sample was step-loaded to 11 ksi for 125 hours, and then, after increasing the load to 12 ksi, the sample failed after 71 hours. A sketch of both test samples is shown in Figure 10. A 2000°F tensile test showed a UTS of 15.0 ksi, a 0.2 % YS of 10.3 ksi, and an elongation of 1.5 %.

Dimensional

Both the elliptical and the vane section had as-extruded surfaces of considerable smoothness. The dimensions of the preform of the vane section were reduced by the factors, which correspond within 10 percent of the anticipated dimensions, arising from the geometric relationships, between the die and the liner. (These relationships, controlling dimensions in the filled billet method, were discussed in the Introduction.) The extruded size was consistently smaller than that expected from a dense preform. A substantial amount of the deviation is presumed to be attributable to the 65 percent relative density of the preform.

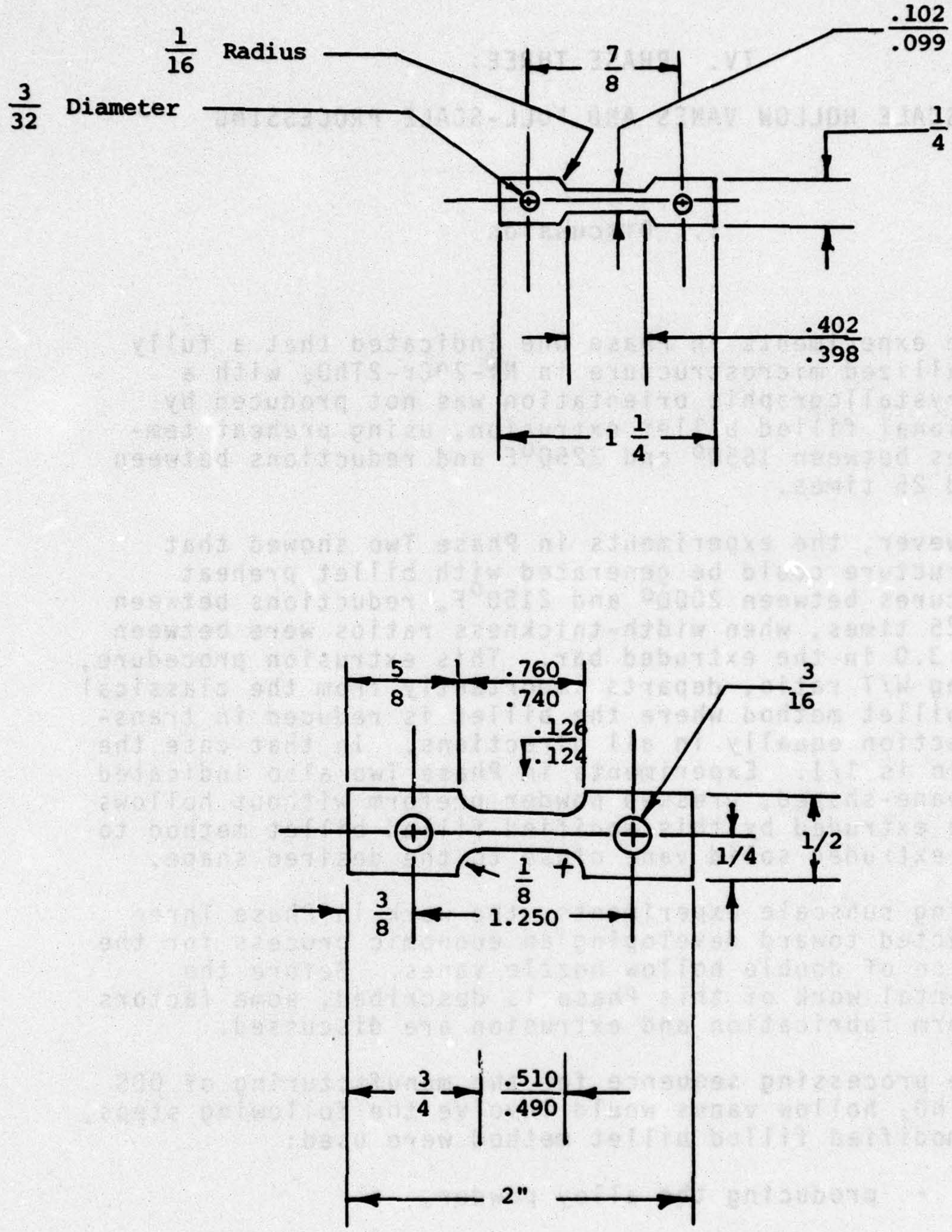


FIGURE 10. SKETCH OF MECHANICAL TEST SPECIMENS IN PHASE TWO

Top: Specimen from elliptical rod (Billet 27).

Bottom: Specimen from solid vane (Billet 28).

IV. PHASE THREE:

SUBSCALE HOLLOW VANES AND FULL-SCALE PROCESSING

1. Discussion

The experiments in Phase One indicated that a fully recrystallized microstructure in Ni-20Cr-2ThO_2 with a <100> crystallographic orientation was not produced by conventional filled billet extrusion, using preheat temperatures between 1650^{o} and 2250^{o}F and reductions between five and 25 times.

However, the experiments in Phase Two showed that this structure could be generated with billet preheat temperatures between 2000^{o} and 2150^{o}F, reductions between 10 and 25 times, when width-thickness ratios were between 2.5 and 3.0 in the extruded bar. This extrusion procedure, involving W/T ratio, departs importantly from the classical filled billet method where the billet is reduced in transverse section equally in all directions. In that case the W/T ratio is 1/1. Experiments in Phase Two also indicated that a vane-shaped, pressed powder preform without hollows could be extruded by this modified filled billet method to form an extruded solid vane close to the desired shape.

Using subscale experiments, the work in Phase Three was directed toward developing an economic process for the production of double hollow nozzle vanes. Before the experimental work of this Phase is described, some factors in preform fabrication and extrusion are discussed.

The processing sequence for the manufacturing of ODS Ni-Cr-2ThO_2 hollow vanes would involve the following steps, if the modified filled billet method were used:

- producing the alloy powder,
- preconsolidating the powder to about two-thirds relative density,
- producing the preform,

- canning the preform in fillers in a billet,
- extruding by the modified filled billet method,
- straightening the extrusion,
- decanning the extruded hollow vane, and, finally,
- heat treating the vane.

Powder production, preconsolidation, canning, decanning, and heat treating are established techniques or processes. The feasibility of extruding by the modified filled billet method was demonstrated in the Phase Two experiments of this program. Straightening is not regarded as a difficult or complex problem.

Apparently, economic production of a precise, hollow vane preform is the step in the manufacturing sequence requiring development. Such preform could be made by hot isostatic pressing or hot axial pressing with refractory tools, followed by various amounts of machining, if necessary. However, this work would be outside the scope of this program.

An important consideration in preform preparation is that the preform ideally should have a length-to-"diameter" relationship similar to a conventional extrusion billet, so that it can be efficiently extruded.

In filled billet work the amount of loss of preform material to "uncontrolled end-effects" during extrusion is more or less fixed. When the length of the preform is about the same as the diameter of the billet, and when higher reductions are used, the end loss is generally quite high, probably about 30 percent. At lower reductions the end loss is lower. The yield loss at higher reductions can be greatly reduced by extending the core length and billet length until the length of the core is about three or more times the billet diameter. Then the loss is about 10 percent or less, even when higher, more efficient reductions are used.

Since the full scale vane for the F101 engine is about 2.6 inches wide, even by the modified filled billet method, the preform should be about six inches wide. In view of efficiency considerations, the full scale preform should be about 18 to 24 inches long.

Making precision hollow preforms by machining away large amounts of dense material is relatively impractical or expensive because of the metal removal cost and the cost of the metal removed. Thus, consideration of methods of preform production should include the concept that the method be capable of producing relatively precise preforms of practical length without machining dense material at all, or at least not in the length near the full billet length.

Another factor in preform production is that consolidation should be carried out under conditions which will not prevent the attainment of the desired recrystallized microstructure that would otherwise be generated by the chosen method of hot extrusion. Appreciable amounts of working of the powder may alter recrystallization behavior.

In view of these considerations, preextrusion was evaluated as a means of producing preforms for subsequent reextrusion. The method has the advantages of having the potential of producing a precision dense shape with sufficient length-to-"diameter" ratio. Green-machined preforms could be used, equipment for preextrusion was available, and the effort was within the scope of work of the program.

Thus, for the purposes of this program, the processing sequence referred to previously to manufacture ODS Ni-Cr-2ThO_2 hollow vanes can be modified to involve the following steps:

- producing the alloy powder,
- isostatically pressing the powder to about two-thirds relative density,
- green machining a double hollow preform from the compact,
- canning the preform in fillers in a billets,
- densifying the preform in the billet, and then in the same or separate step,
- preextruding by conventional filled billet method,

- reextruding by modified filled billet method,

- straightening the extrusion,

- decanning the extruded hollow vane, and, finally,

- heat treating the vane.

The added steps of densifying and preextruding are included in the investigations in Phase Three.

2. Scope

The experiements in Phase Three first involved the evaluation of three contemplated methods of densifying powder preforms. The preforms made in the densification trials were ultimately preextruded and then reextruded into finished hollow vanes by the modified filled billet method.

The recrystallization behavior of some of these extruded hollow vanes was examined.

Following this work, the best method of preform densification was used to make a group of four hollow preforms and two solid preforms with the same external dimensions. These preforms were preextruded by the conventional filled billet method and reextruded by the modified filled billet method to determine the reproducibility of the process. The conditions chosen for reextrusion were based on the best results obtained in Phase Two. After heat treatment, samples of the extruded hollow vanes were examined metallographically. Samples of the solid vane produced under the same conditions were also examined metallographically, and tested in dynamic oxidation tests and stress rupture tests.

Finally, the information generated in this work was formulated into a preliminary specification for the full scale extrusion of double hollow vanes. This work completed Phase Three and the program.

3. Procedure

Evaluation of Preform Densification Methods

Compact Pressing

Preforms for the three densification methods were made from the lot of powder described in Phase One. A 20-pound portion was isostatically pressed at 33 ksi into a block 3 by 3 by 10 inches. A solid vane shape, or single-hollow vane shape could have been pressed to save powder and machining, but special tools would have been required.

Compact Machining

All three preforms for the three experimental densifications were "green" machined. The outside contour was obtained by band sawing; the double-hollow contour by drilling and band sawing. Local irregularities were removed by filing. The machined preforms were about 2.5 inches long and 2.5 inches wide. They had the same external shape with two parallel hollows. The first two had walls about 0.2-inch thick, and the other had walls about 0.1-inch thick.

The pressing had machining characteristics similar to graphite. Experience indicated that green preforms of practical length could be obtained with a dimensional tolerance of ± 0.015 inch. The chips generated could be kept clean, and was apparently unworked, and therefore probably capable of being recycled.

Compact Densification

All three green-machined, hollow preforms were assembled in fillers and canned in a thin sleeve of steel. The assembly comprised billets that were evacuated and then consolidated in a 3.070-inch liner at 2000°F and 100 tsi, as indicated in Table 3.

The first thick-walled preform (in Billet 33) was surrounded with filler consisting of free-flowing steel powder with a tap density of about 65 percent, close to the

TABLE 3

PREFORM DENSIFICATION CONDITIONS AND RESULTS IN PHASE THREE

	Conditions				Results
Billet No.	Billet Design (Figure 10)	Preform Wall Thickness (in.)	Steel Filler Density (%)	Billet Components Receiving Initial Axial Compression	Shape Retention
33	Top	0.2	65	Filler and preform	Fair
34	Middle	0.2	100	Filler and preform	Fair
35	Bottom	0.1	100	Preform only	Good

Other Conditions:

Preheat Time	2 hours
Preheat Temperature	2000° F
Compacting Pressure	100 tsi
Compacting Time	15 seconds

relative density of the alloy preform. Compression was applied simultaneously across the entire cross section of the alloy and steel powder. Since the alloy and steel powder densities were close, more or less uniform axial compression was expected.

The second thick-walled preform (in Billet 34) was similar in shape to that in Billet 33 and was surrounded with dense steel fillers. Compression was axially applied across the entire cross section like Billet 33.

The third preform, the one with a thin wall, was enclosed in a billet (Billet 35) which was much like Billet 34. However, the axial compression was applied only on the cross section of the alloy preform with the use of mild steel "penetrators" that extended beyond the length of the fillers. See Figure 11.

The three compacted preforms were evaluated by comparing their dimensional uniformities, their capabilities for subsequent extrusion and their subsequent recrystallization behavior.

Dimensional Uniformity

The three consolidated billets containing the preforms were turned on a lathe to remove about one-half inch from the diameter of the upset billets, and faced to expose the cross section of the preforms at both ends. The billets were then x-rayed to reveal the preforms' relative longitudinal uniformity, and examined at the exposed ends to observe the preforms' cross-sectional shape retention. The lengths of the dense preforms were about two inches.

The dense preform in Billet 33, compacted in powder, showed substantial bowing, fair cross-sectional shape retention, but thickened walls. The steel was removed from this preform by pickling in nitric acid. The bare preform is shown in Figure 12.

The dense preforms, consolidated in solid fillers (Billets 34 and 35), lacked the bowing seen in the preform from Billet 33. The ends of the preform in Billet 34 had shape retention similar to Billet 33, but better wall thickness stability.

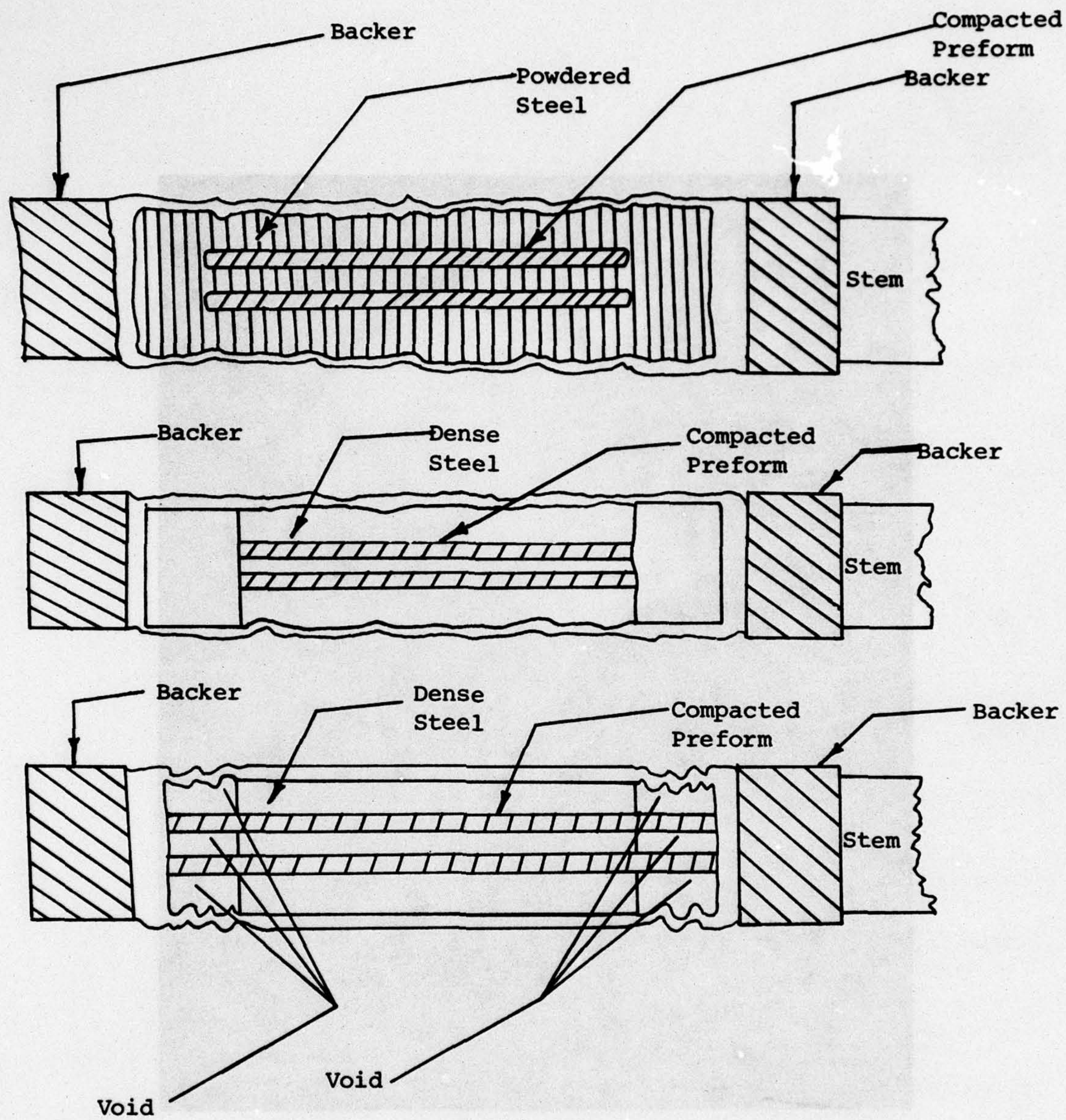


FIGURE 11. SKETCH OF BILLET DESIGNS FOR PREFORM DENSIFICATION IN PHASE THREE

Top: Billet 33 - Compaction of powdered steel and preform.

Middle: Billet 34 - Compaction of dense steel and preform

Bottom: Billet 35 - Compaction of preform

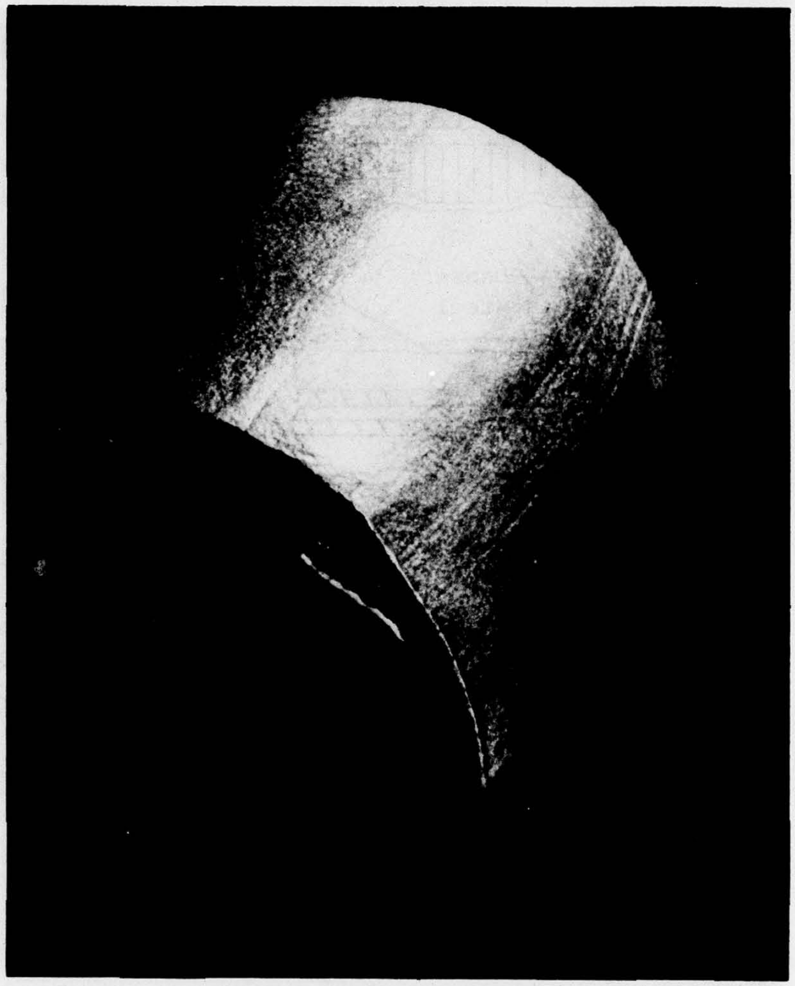


FIGURE 12. PHOTOGRAPH OF PREFORM DENSIFIED IN POWDER IN PHASE THREE

When a powder filler is used, considerable bowing of the preform occurs on densification (Billet 33). (Scale about 1.5X)

The cross-sectional shape retention of the preform consolidated by penetrators (in Billet 35) was markedly superior to the other preforms, even though a much thinner-walled preform was attempted.

Extrusion Capability

The preforms (in Billets 34 and 35) made in solid steel were preextruded at 2000°F and three times reduction to produce a core for reextrusion with an L/D of about 5 to 1. The cross section of the preextruded cores, revealed by cropping, were found to be satisfactory. A photograph of the cross section at the fronts of these preextrusions is seen in Figure 13. The longitudinal variation was found by x-ray examination to be minimal.

The reextrusion characteristics of the preform from Billets 34 and 35 were evaluated by reextruding these preforms with steel fillers in place in a can at 2000°F, and 10 times reduction through a die producing a rod with a width-thickness ratio of 2.5 to 1. These conditions produced good results in Phase Two.

The extrusions were cropped and then decanned by pickling in nitric acid. Figure 14 shows a photograph of the thin-walled hollow vane section (from Billet 35). A larger, solid vane, produced in Phase Two from Billet 24, is included for comparison.

Recrystallization Behavior

The double-extruded preforms were recrystallized and metallographically examined by the methods described in Phase One. The first sample taken from the front of extrusion 35 was found almost entirely to be made up of the desired <100> texture. However, two small areas, comprising less than 10 percent of the cross section, contained the <110> texture and unrecrystallized material previously observed in earlier work. Another sample nearer the center of the extrusion was recrystallized and found to contain only recrystallized <100> grains.

These experiments indicated a process outline for making double hollow vanes with the desired properties. In succeeding work the reproducibility of the process was evaluated.

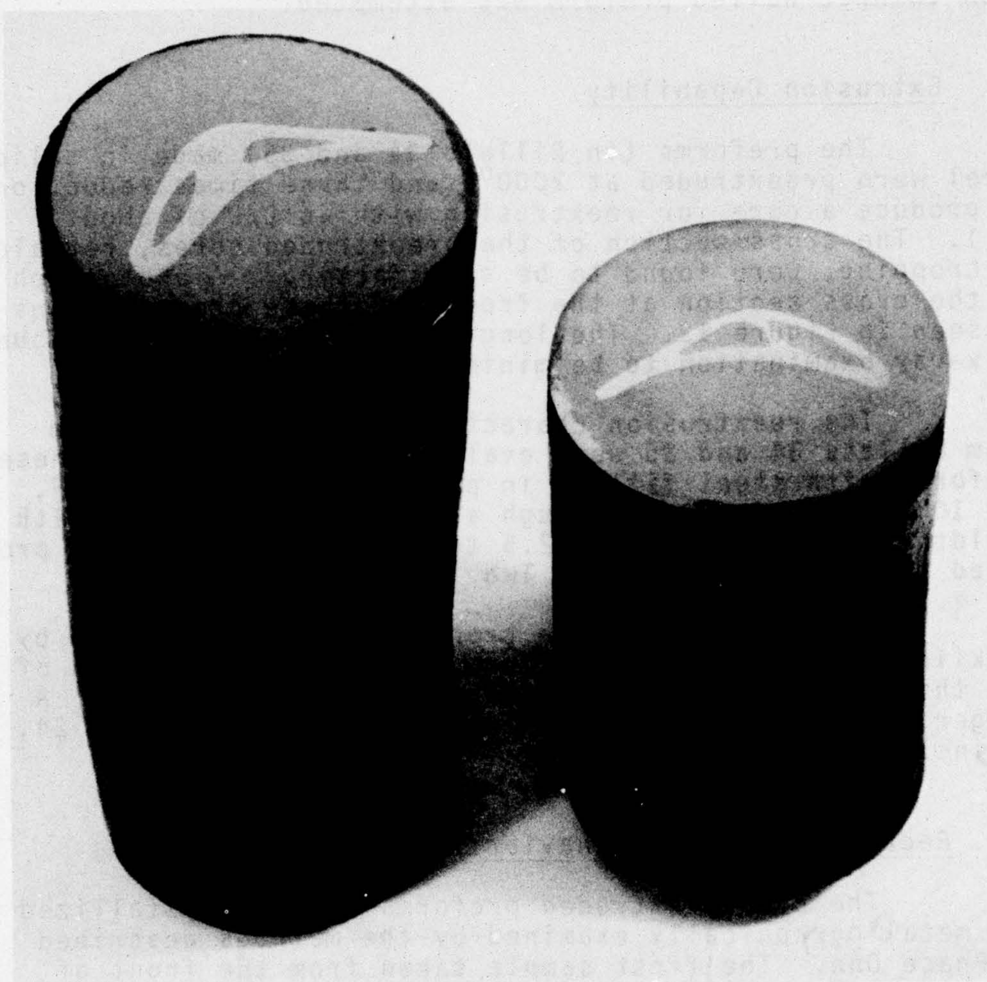


FIGURE 13. PHOTOGRAPH OF CROSS SECTIONS OF PREEXTRUDED PREFORMS IN PHASE THREE.

Preextrusion provides an efficient means of lengthening a preform. (Left, extrusion from Billet 34; Right, extrusion from Billet 35.) Scale about 1.5X

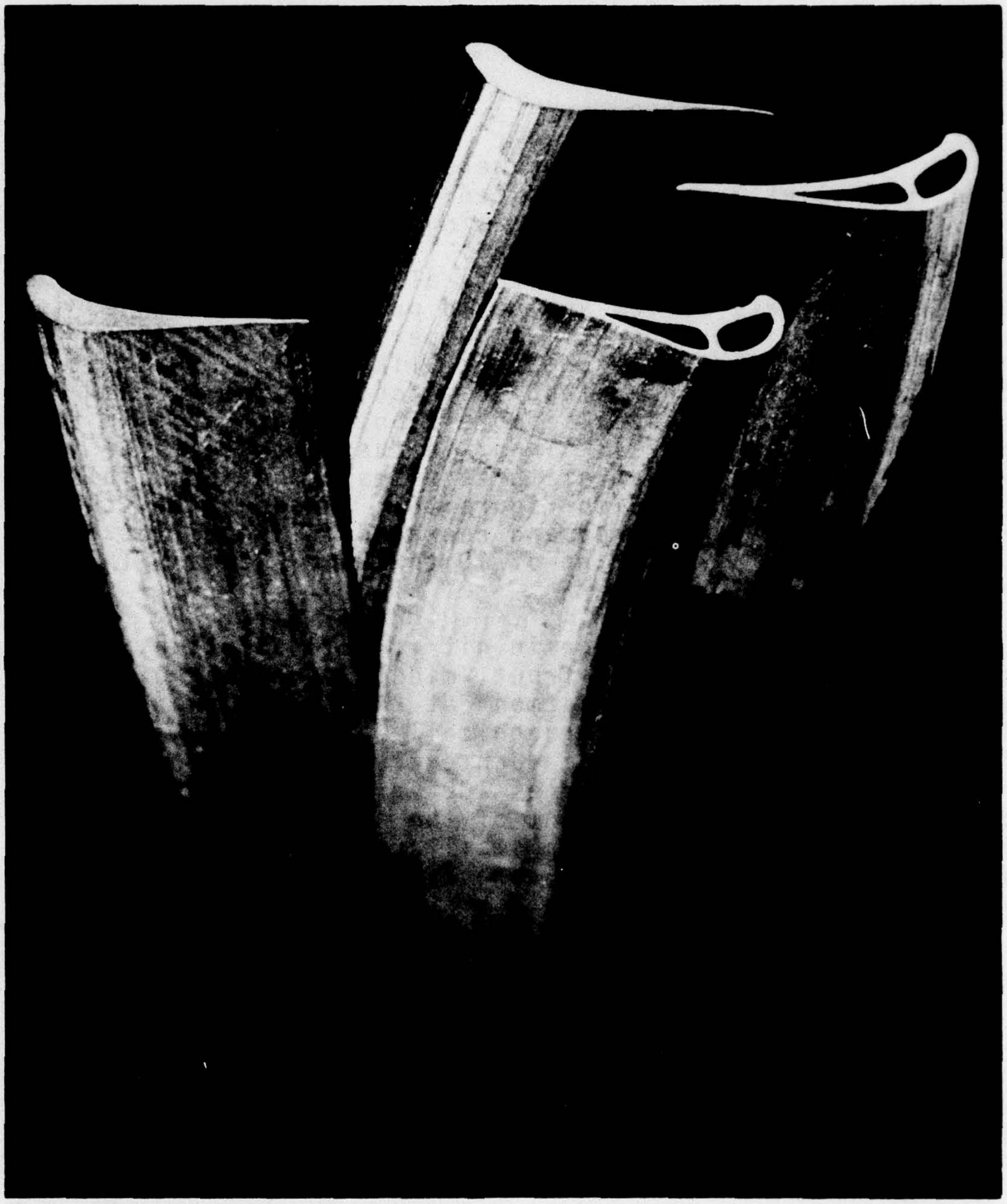


FIGURE 14. PHOTOGRAPH OF HOLLOW AND SOLID VANES

Reextrusion of a double hollow-vane preform (from Billet 35). Two solid vanes (Billet 24) are shown for comparison. (Scale about 3X)

Evaluation of Extrusion Reproducibility

Sample Preparation

Five hollow preforms were prepared like the one used for Billet 35 which produced the best results in previous work. A sketch indicating some of the preform dimensions is shown in Figure 15. Two solid preforms with the same external dimensions were also made by the same technique. These hollow and solid preforms were canned in steel, and assigned the billet numbers 36 through 42, respectively. They were compacted like Billet 35 but at 2000°F, 50 tsi, by the method in which only the preform receives the initial axial compression, illustrated in the bottom view of Figure 11. Then, in the same step, the billets were preextruded at three times reduction. This sequence is illustrated in Figure 16. Subsequently, the extrusions were remachined and reextruded like Billet 35 at 2000°F, with a reduction of 10 to 1 and with a W/T ratio of 2.5 to 1.

Following pickling in nitric acid solution, samples were taken at the front, middle, and rear of each extrusion. After heat treatment at 2450°F for two hours, they were examined metallographically. All the samples from the middle and rear of each extrusion were found to recrystallize with only large <100> grains. However, two of the samples from the front of the hollow vanes, extrusions 37 and 38, contained a small area with some grains with <110> orientation. No unrecrystallized material was observed.

Dimensional Uniformity

The front, middle and rear samples, used for metallographic examination above, were measured in the width and thickness directions. The location of the partition between the front and rear hollow was also measured in the hollow vanes. These results are listed in Table 4. The theoretical dimensions anticipated from the extrusion reduction of the preform also are listed. The departure from theoretical is under seven percent. Views of typical transverse cross sections are seen in Figure 17.

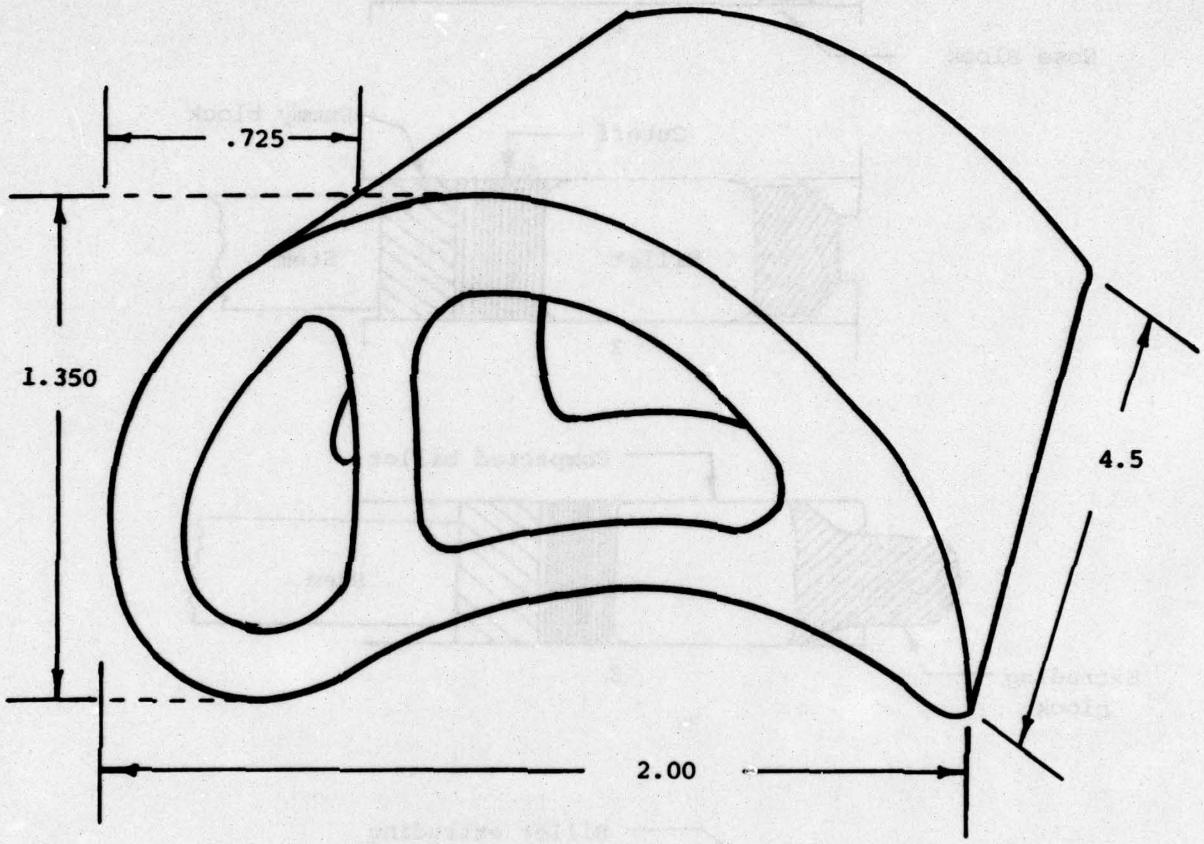


FIGURE 15. SKETCH OF PREFORM

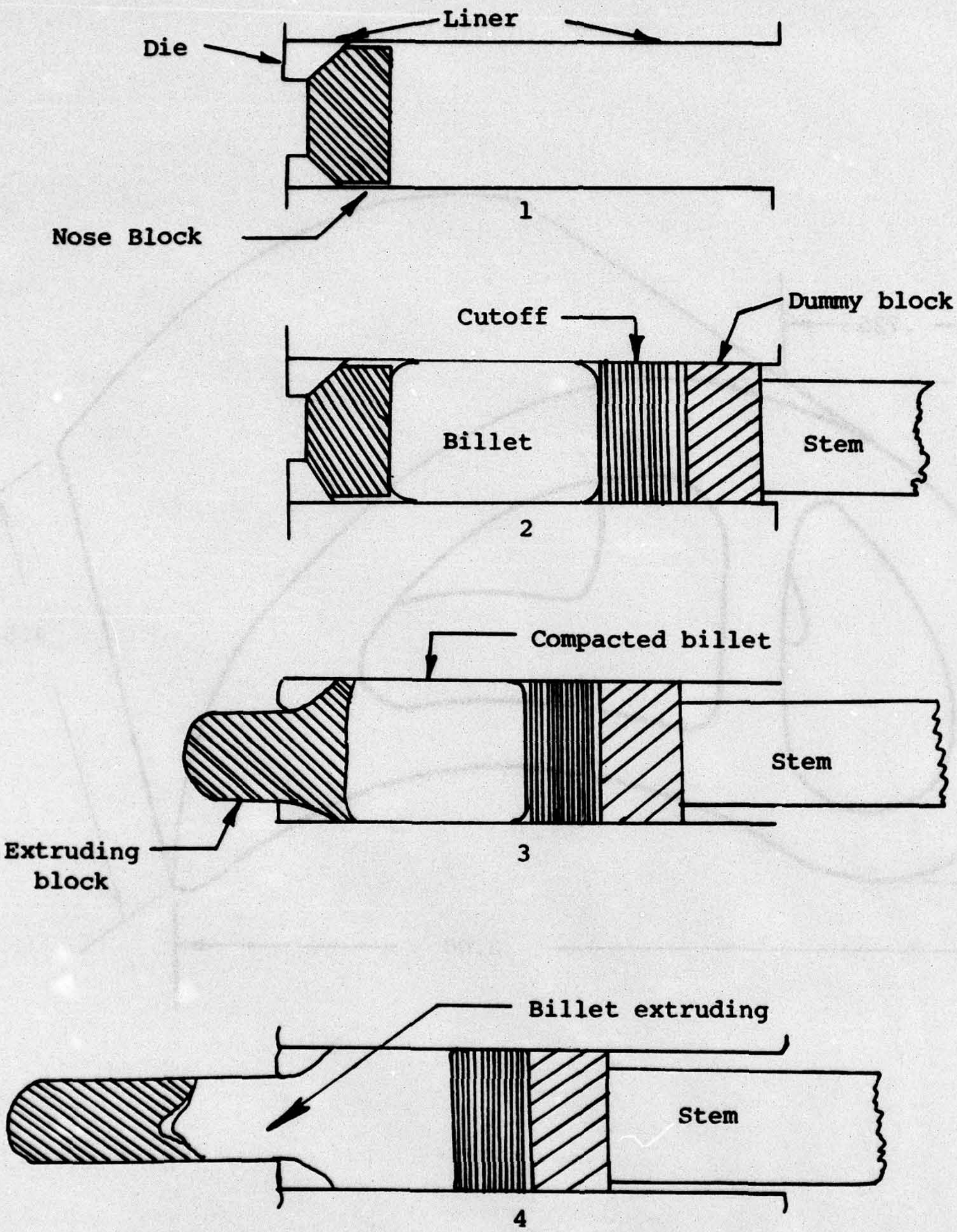


FIGURE 16. COMPACTION AND PREEXTRUSION

1. Stiff nose block (steel at 1200°F) in liner
2. Billet, cutoff and dummy block in liner
3. Nose block extruding, billet compacting
4. Billet extruding

TABLE 4

DIMENSIONS OF EXTRUDED VANES [1]

Extrusion Number	Measurement	Actual [2] Front	Middle	Rear
36	width	.830	.809	.790
	height	.112	.110	.106
	partition	.166	.170	.178
37	width	.810	.795	.760
	height	.106	.109	.101
	partition	.162	.159	.152
38	width	.821	.803	.788
	height	.111	.108	.098
	partition	.169	.163	.158
39	width	.818	.803	.781
	height	.113	.109	.106
	partition	.164	.158	.147
40	width	.840	.836	.820
	height	.126	.132	.138
	partition	solid	solid	solid
41	width	.838	.830	.826
	height	.121	.127	.129
	partition	solid	solid	solid

[1] Theoretical values are .860, .129 and .170 for the width, height, and partition position, respectively. The latter is the distance from the leading edge to the partition.

[2] Measured at 5, 15 and 25 inch positions on 30 in. specimens.

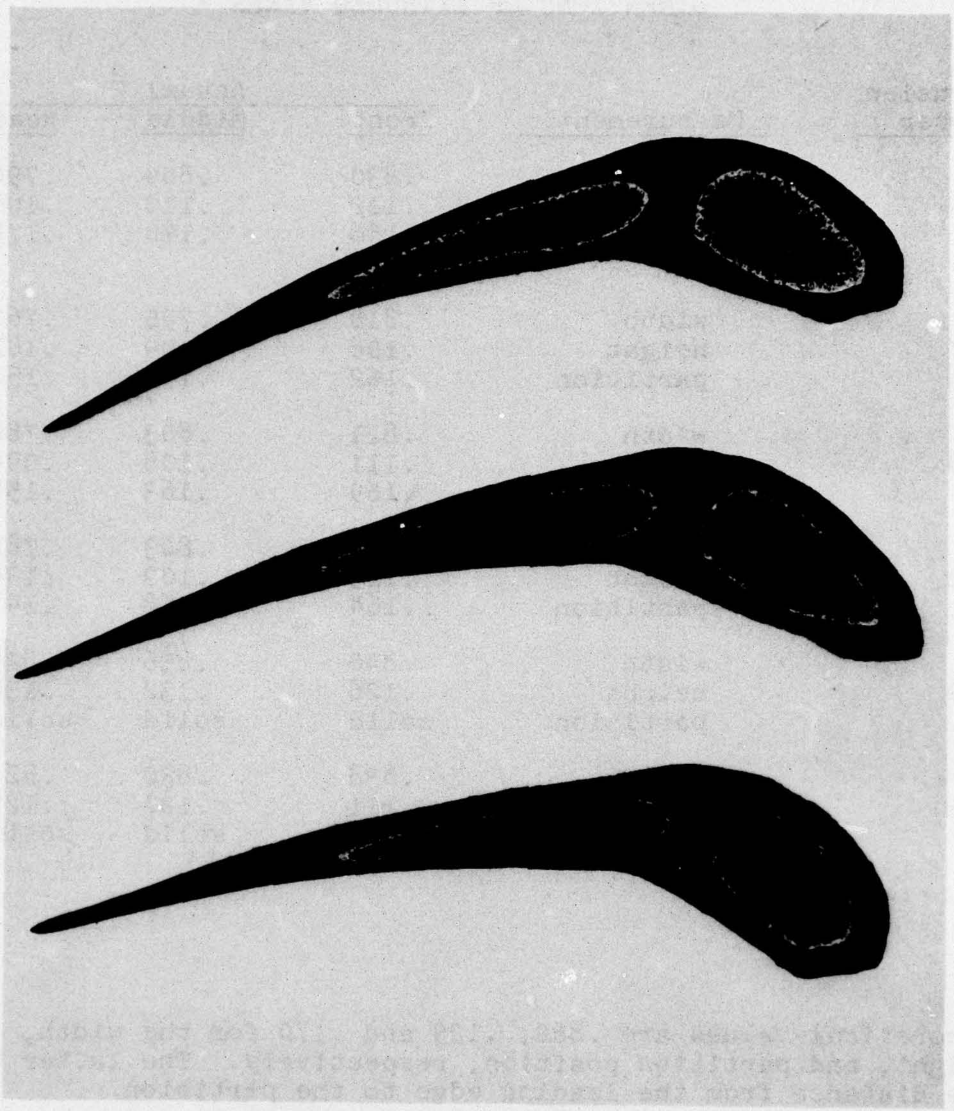


FIGURE 17. CROSS SECTION OF EXTRUDED HOLLOW VANE

Three transverse cross-sections of Extrusion 39 and the front, rear and middle, respectively. The extrusion was about 30 inches long. The samples were taken at the 5, 15 and 25 inch portions. Magnification is about 6X. Steel is present in the hollows.

Dynamic Oxidation Testing

The solid vane extrusion from Billet 41 was used for these tests, since the hollows from Billets 36 through 40 appeared to be too fragile.

The dynamic oxidation and hot corrosion tests were performed in an engine environmental simulator or burner rig by Sherritt Gordon Mines Ltd. The burner consists of a can-type combustor with fuel atomization from a single nozzle and a converging-diverging exit nozzle with a 0.8 inch throat diameter. The mass flow rate through the burner was approximately 200 lb/hr and the fuel (JP 4)-to-air ratio (by weight) varied from 1:29 at 2000°F to 1:15 at 2200°F. The resulting combustion gas velocity at the exit nozzle was approximately Mach 0.3. The burner was fueled with commercial JP 4 containing 0.026 w/o sulfur.

Four tests were tried. Three 100-hour tests at temperatures of 2100°, 2200°, and 2325°F were completed. However, the fourth test after 65 hours at 2250°F was discontinued because of iron contamination in the center of the test specimens, not noted prior to starting the test.

The dynamic oxidation data, listed in Table 5, are reported as the weight loss per unit area (mg/cm^2). This weight was calculated on an exposed surface area and did not include that portion of the sample which was clamped in the holder. Dynamic oxidation data also are included from wrought sheet samples of Ni-20Cr-2ThO_2, which were used with the vane samples in each test as a standard.

A comparison of the data from the two materials shows a higher weight change per unit area for the vane samples than for the wrought sheet samples. Apparently, this is due to the larger grain size present in the vane material than in wrought sheet samples.

The vanes were observed after various times in each oxidation test. Indication of cracks in the material were not seen. Although the cyclic dynamic oxidation test does not simulate a proper thermal fatigue test, the material tested would appear to have good thermal fatigue resistance.

TABLE 5

CYCLIC DYNAMIC OXIDATION TESTING OF EXTRUDED VANES

Test Temperature 2100^0F

Sample[1]	Weight Loss, mg/cm^2 16.5 (hr)	47.0 (hr)	71.0 (hr)	87.0 (hr)	100.0 (hr)
1	0.7	2.0	3.2	4.5	5.5
2	0.8	2.1	3.4	4.9	*
3	0.8	2.6	4.0	5.2	*
4	0.7	2.6	4.2	*	*
$NiCrThO_2$ Sheet (ave)	1.0	2.0	3.4	4.0	5.0

*Samples broke in burner rig

[1] Samples taken from Extrusion 41

Test Temperature 2200^0F

Sample	Weight Loss, mg/cm^2 20.0 (hr)	40.0 (hr)	50.0 (hr)	60.0 (hr)	70.0 (hr)	90.0 (hr)	100.0 (hr)
1	3.6	7.4	10.0	12.0	13.6	16.0	19.0
2	1.4	4.4	5.2	6.8	8.4	10.0	12.0
3	2.4	5.6	7.0	9.2	11.0	12.4	15.2
$NiCrThO_2$ (ave)	1.2	3.2	3.5	5.0	6.0	10.0	13.0

Test Temperature 2325^oF

Sample	Weight Loss, mg/cm^2 14.0 (hr)	32.0 (hr)	46.0 (hr)	54.0 (hr)	64.0 (hr)	71.0 (hr)	82.0 (hr)	91.0 (hr)	100.0 (hr)
1	3.0	17.0	44.0	56.0	73.0	88.0	104.0	128.0	146.0
2	4.0	25.0	46.0	60.0	80.0	96.0	112.0	132.0	157.0
3	4.0	30.0	50.1	65.0	86.5	100.0	120.0	140.0	160.0
4	4.5	28.0	51.0	70.0	92.0	108.0	124.0	145.0	168.0
$NiCrThO_2$ Sheet (ave)	3.5	8.0	18.0	24.0	30.0	38.0	40.0	48.0	58.0

Dispersion Size and Distribution

Transmission electron photomicrographs were taken from a selected area near the center of the extruded solid vane section from Billet 41. The photomicrographs shown in Figures 18A, 18B and 18C are at magnifications of 10,000, 20,000 and 30,000 times, respectively. Dispersoid size and distribution are similar to that observed in our Ni-20Cr-2ThO_2 wrought sheet.

Mechanical Testing

Prior to stress rupture and tensile testing, samples were given two types of recrystallization treatment. The first was a furnace recrystallization mentioned previously: 2450^oF for 2 hours. The specimens were loaded at 2200^oF on an alumina refractory, and heated to 2450^oF in 30 minutes. After two hours, the specimens were removed from the furnace and air cooled.

The second type of heat treatment was directional recrystallization. In this case the samples were vertically lowered into a salt bath at 2450^oF at a controlled rate of 0.6 inches per minute. The thermal gradient was estimated to be 2000^oF/in.

Initially, difficulty was encountered with the hollow vane specimens. Due to oxidation of the thin walls during this heat treatment, little material was left for metallographic examination or mechanical testing. Therefore, mechanical testing was completed on samples of solid vanes.

As shown in Table 6, which lists the results of stress rupture and tensile tests, furnace recrystallized material, when previously extruded under the proper conditions, had 2000^oF stress rupture and tensile properties which exceeded the objectives of the program. These target values were 100-hour stress rupture life at 10 ksi, and 10 ksi tensile strength. Although directional recrystallization caused improvement in stress rupture and tensile properties, this technique was used on a few specimens, because furnace recrystallization met the requirements of the program.

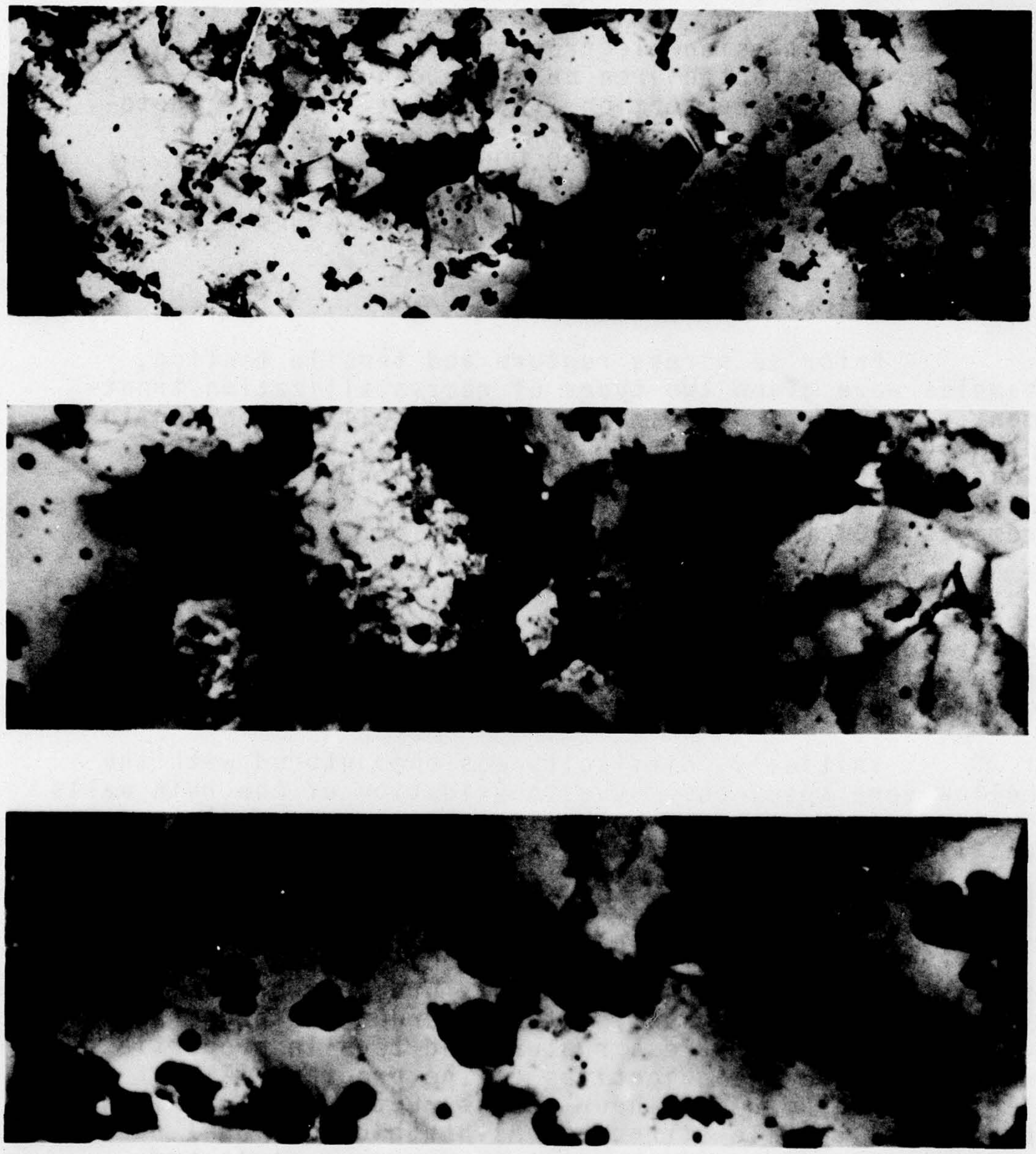


FIGURE 18. DISPERSOID SIZE AND DISTRIBUTION

Photomicrographs at: A - 10,000X
B - 20,000X
C - 30,000X

TABLE 6

MECHANICAL TESTS[1] ON EXTRUDED VANES

$2000^{o}F$

Extrusion No.	Heat Treatment	Sample Position	Stress Rupture Life 10 ksi (hr)	Stress Rupture Life 11 ksi (hr)	Tensile Strength Ultimate (ksi)	Tensile Strength Yield (ksi)	Tensile Strength Elongation (%)
24	Furnace	Front	-	-	14.3	9.8	1.1
		Middle	240[2]	125	15.0	10.3	1.5
		Rear	-	-	16.1	11.2	1.6
	Directional	Front	-	142	17.8	17.2	8.6
		Middle	250[2]	165	18.0	16.2	9.4
		Rear	-	157	17.0	15.8	7.4
42	Furnace	Front	-	138	17.3	15.4	1.2
		Middle	240[2]	159	16.5	16.2	1.4
		Rear	-	161	14.9	14.6	1.3
	Directional	Front	-	180	15.6	14.2	7.6
		Middle	240[2]	171	19.1	17.6	9.2
		Rear	-	150	16.3	15.8	8.1

[1]Testing parallel with the extrusion direction
[2]Then step loaded to 11 ksi

As mentioned previously, the solid vane extrusion No. 41 was used for the oxidation tests. The other solid vane extrusion from Billet 42, extruded under the same conditions as Billet 41 and the hollow vane Billets 36, 37, 38 and 40, provided samples for mechanical testing in Phase Three. The furnace recrystallized microstructure of Extrusion 42 closely matched that of the hollow vane extrusions. A very similar microstructure, except probably with larger grains, was produced by directional recrystallization. The grain length was difficult to measure because the grain boundaries had a highly irregular shape that appeared to rise and fall out of the plane of the polished section.

Samples were taken from the front, middle and rear of Extrusion 42. After heat treatment they were machined to the dimensions shown in Figure 10, bottom. The results of the 2000°F tensile and stress rupture tests are shown in Table 6. Test results from Phase Two, involving Extrusion 24, are included for comparison.

4. Process Specifications for Full-Scale Nozzle Vanes

The experimental work completed in Phase Three of this program provides the basis for a preliminary process specification for full-scale nozzle vanes of ODS Ni-20Cr-2ThO_2. The production of full scale vanes would involve the following steps:

Powder Procurement

A production quantity of ODS Ni-20Cr-2ThO_2 would be procured to a commercial specification.

Powder Pressing

The powder would be converted to extrusion preforms by isostatic pressing followed by machining. The pressing conditions would involve about 33 ksi and 30 second dwell time. The shape of the pressing could be a solid rectangular block, a solid vane, a single hollow vane, or a double hollow vane. The choice would be determined by the quantity of preforms produced, since greater production amounts would justify the increased tooling cost for pressing shaped and hollow preforms.

Preform Machining

Following pressing, the preform would be machined by band sawing without lubricant. If the hollows are not formed in the previous pressing step, it would be formed in this step by dry drilling, followed by contour band sawing. The length of the preform would be close to its width, about six inches. The dimensional tolerances in the preform would be improved by filing. Scrap powder or pieces would be retained for recycling.

The design of the transverse cross section of the preform would be controlled by the chosen extrusion ratios of preextrusion and reextrusion, and by the W/T ratio of the reextrusion. The dimensions of the preform can be calculated from the final or desired shape. The details of this calculation are proprietary as they related to proprietary tooling.

Billet Design and Assembly

The billet would consist of eight parts: a nose, tail, can, inner fillers, outer fillers, front and rear penetrators, and preform. Except for the preform these parts would be made of low carbon steel. The nose and tail could be cut from bar or made from flame-cut "burnouts". The can could be seamless or welded pipe or tubing. The inner and outer fillers could be extruded steel shapes or a stack of shaped "burnouts" or stampings. The latter forming method could be used to make the penetrators. The preform would be made by machining a green pressing, as mentioned earlier. The shape of the fillers would be such that the volume inside the preform is filled with inner filler and the volume between the preform and can is filled into the outer filler. The tail and nose, when inserted into the can, make up a cylindrical assembly. Before assembly each steel component would be cleaned. A simple means of evacuating and sealing the billet would be provided by a small tube through the nose or tail.

Assuming the preform after densification is eight inches long, with two-thirds relative density, the preform would be 12 inches long before densification. During this process the penetrators would travel two inches each, and the overall billet length would be about 25 inches.

Densification and Preextrusion

After preheating to 2000°F, the billet would be loaded into the liner of the extrusion press. A liner about nine to ten inches in diameter would be used. A colder "nose block" would have been previously placed in the liner. As the force builds up to extrude the stiff nose block, the green preform would be densified by the travel of the penetrators. Then the nose block would be extruded followed by the billet, as shown in Figure 16. The extrusion ratio chosen would be close to three times, so that the preextruded preform would be now about 24 inches long.

Reextrusion

Since the preextrusion die is chosen so that the primary extrusion is just under the liner size for the reextrusion, very little machining would be necessary to prepare the billet for this step.

Cropping at the front and rear of the extrusion would be carried out. Following preheating to 2000°F, the billet would be extruded in a direction opposite that of the primary extrusion to maintain yield at a higher level. Extrusion would take place between 125 and 150 inches/minute. A force of about 1900 tons would be expected.

Straightening

If the extrusion is received by a runout tube at the extrusion press, little, if any, straightening would be necessary. This is especially true since the extrusion would be cut into relatively short lengths in subsequent steps.

Decanning

After cropping off excess steel from the front and rear of the extrusion, and grit blasting the extrusion to remove oxide, the coextruded canning and filler on the outside of the preform would be removed with nitric acid solution. Approximately one-third gallon of 70-percent nitric acid solution is needed to remove each pound of steel. Then the extrusion would be cut into convenient lengths for subsequent acid removal of the inner filler.

Heat Treatment

Before final machining, the decanned sections would be given a recrystallization heat treatment. The furnace heat treatment might be adequate: 2450^{o}F for 2 hours. Alternatively, a directional recrystallization could be used.

V. CONCLUSIONS

The experimental program demonstrated that the filled billet extrusion method, when suitably modified, can produce double hollow nozzle vanes with net or nearly net dimensions and with high temperature properties comparable to sheet or to gross extrusions of the same alloy.

The process, when carried out on full scale vanes, should enable considerable cost reduction since significantly less material and machining would be required to produce a finished vane. These savings are believed to be considerably greater than additional processing cost arising from the use of the filled billet method.

This process should be applicable to similar oxide dispersion strengthened alloys such as the advanced mechanical alloys.

The most significant finding of the program was the relation of the width-thickness ratio of the extruded bar to the recrystallized microstructure.

VI. RECOMMENDATIONS

At this writing, the alloy Ni-20Cr-2ThO$_2$ has been displaced by more advanced oxide dispersion strengthened alloys. Cost reduction in the production of vanes of these alloys should be achieved if the modified filled billet method were used.

For this reason, a full-scale development of vane extrusion of one or more of the alloys by the modified filled billet method is recommended. This work should be preceded by successful subscale work to verify the attainment of high temperature properties in these alloys by this new method.

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